PILOT PROJECT QUALITY ASSURANCE PROJECT PLAN

WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE WAUKEGAN, ILLINOIS

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QUALITY ASSURANCE PROJECT PLAN

PROJECT TITLE: PILOT PROJECT
QUALITY ASSURANCE PROJECT PLAN
Waukegan Manufactured Gas and Coke Plant Site
Waukegan, Illinois

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LIST OF ACRONYMS AND SHORT FORMS

CPT	-	Cone Penetrometer Testing
CRA	-	Conestoga-Rovers & Associates
DQO	-	Data Quality Objective
DO	-	Dissolved Oxygen
EC	-	Electrical Conductivity
GC/MS	-	Gas Chromatography/Mass Spectrometry
ICP	-	Inductively Coupled Plasma
ICS	-	Interference Check Sample
IEPA	-	Illinois Environmental Protection Agency
LCS	-	Laboratory Control Sample
LCS/DUP	-	Laboratory Control Sample/Laboratory Duplicate
LCS/LCD	-	Duplicate Laboratory Control Sample Analyses
MS/DUP	-	Matrix Spike/Laboratory Duplicate
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
ORP	-	Oxidation-Reduction Potential
%R	-	Percent Recovery
PAHs	-	Polynuclear Aromatic Hydrocarbons
QA	-	Quality Assurance
QA/QC	-	Quality Assurance/Quality Control
QAPP	-	Quality Assurance Project Plan
QC	-	Quality Control
RI/FS	-	Remedial Investigation/Feasibility Study
ROD	-	Record of Decision
RPD	-	Relative Percent Difference
RPM	-	Remedial Project Manager
SAP	-	Sampling and Analysis Plan
Site	-	Waukegan Manufactured Gas and Coke Plant Site
SOPs	-	Standard Operating Procedures
SRM		Standard Reference Material
U.S. EPA	-	United States Environmental Protection Agency
UVF	-	Ultraviolet Fluorescence
VOCs	-	Volatile Organic Compounds
WCP	-	Waukegan Manufactured Gas and Coke Plant

1.0 PROJECT DESCRIPTION

This Quality Assurance Project Plan (QAPP) supports the Pilot Project Work Plan (Work Plan) developed by NewFields, Inc. dated May 23, 2000 for the Waukegan Manufactured Gas and Coke Plant (WCP) Site located in Waukegan, Illinois (Site). The Work Plan was developed by NewFields, Inc. on behalf of the WCP Group to address the requirements contained in the Record of Decision (ROD) issued for the WCP Site in September 1999. The Work Plan is reproduced in Appendix A.

The Work Plan was designed to address the Phase 1 elements of the groundwater remedy:

1) short-term groundwater removal and on-Site treatment/re-infiltration; and
2) groundwater treatment.

In addition to the Work Plan and QAPP, other supporting documents have been prepared to assist in the overall goal of successfully completing the Pilot Project. These additional project plans consist of the following documents:

- Health and Safety Plan,
- Sampling and Analysis Plan (SAP),
- Site Management Plan, and
- Treatability Protocols.

1.1 SITE BACKGROUND

The Site background is provided in Section 1.0 of the Work Plan provided in Appendix A.

1.2 SAMPLING NETWORK AND RATIONALE

The sampling network and rationale is summarized in Section 5.0 of the Work Plan provided in Appendix A. This QAPP provides the quality assurance/quality control

(QA/QC) objectives for all samples collected during the Pilot Project except those associated with the bench-scale groundwater treatment assessment described in Section 6.0 of the Work Plan. The analyses and QA/QC objectives associated with the bench-scale groundwater treatment assessment are described in the Treatability Protocols document.

1.3 PROJECT OBJECTIVES AND SCOPE

As stated in the ROD, the design and implementation of the selected groundwater remedy (i.e., the mobile, cell-based, low-flow extraction/treatment/re-injection system) will be based on the current Remedial Investigation/Feasibility Study (RI/FS) data, the pre-design investigation, and pilot testing. Therefore, the objectives of the Pilot Project have been designed to obtain the necessary data required to fully evaluate the implementation, design, operation, and effectiveness of the proposed extraction/re-injection unit of the groundwater remedy specified in the ROD. To meet the objectives of the Pilot Project, the following data collecting activities will be completed:

- pilot study area characterization program,
- pilot extraction and re-injection testing program,
- pilot project system monitoring,
- bench-scale groundwater treatment assessment, and
- pilot project data analysis.

Specific details on the scope of work for these data collecting activities are provided in Section 2.0 of the Work Plan provided in Appendix A.

1.4 PARAMETERS TO BE TESTED AND FREQUENCY

The parameters and frequency of testing are presented in Table 1.1. The selection of these parameters is discussed in Section 5.3 of the Work Plan provided in Appendix A. The laboratory performing the analyses is identified in Section 2.0.

1.5 PROJECT SCHEDULE

The project schedule is provided in Section 9.0 of the Work Plan provided in Appendix A.

1.6 DATA QUALITY OBJECTIVES

The Data Quality Objective (DQO) Process is a series of planning steps based on the Scientific Method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for their intended application.

DQOs are qualitative and quantitative statements derived from outputs of each step of the DQO Process that:

- clarify the study objective;
- define the most appropriate type of data to collect; and
- determine the most appropriate conditions from which to collect the data.

The DQOs were used to develop a scientific and resource-effective sampling design. The DQO process allows decision makers to define their data requirements and acceptable levels of decision during planning before any data are collected. DQOs are based on the seven step process described in "Guidance for the Data Quality Objectives Process", EPA QA/G-4, September 1994.

There are seven steps in the DQO process which include:

- Step 1: State the Problem - The contamination problem that will require new environmental data is summarized, and the resources available to resolve the problem are identified.

- Step 2: Identify the Decision - The decisions that require new environmental data to address the contamination problem are identified.

- Step 3: Identify the Inputs to the Decision - The information needed to support the decision is identified, and which inputs require new environmental measurements are identified.

- Step 4: Define the Study Boundaries - The spatial and temporal aspects of the environmental media that the data must represent are identified.

- Step 5: Develop a Decision Rule - A logical "if...then" statement that defines the conditions that would cause the decision maker to choose among alternative actions is developed.

- Step 6: Specify Limits on Decision Errors - Acceptable limits on decision errors, which are used to establish goals for limiting uncertainty in the data, are specified.

- Step 7: Optimize the Design for Obtaining Data - The most resource-efficient sampling and analysis design for generating data that are expected to satisfy the DQOs is specified.

Using the DQO process ensures that only the data needed to support the project are obtained. The DQOs for this project were determined during the development of the Work Plan (see Appendix A). Table 1.1 summarizes the sampling and analysis program developed for the Pilot Project.

In general, all groundwater and pilot treatment system samples will require off-Site analysis using standard operating procedures (SOPs) based on methods referenced in Section 7.0. Field measurements associated with groundwater and tap water monitoring will be conducted according to SOPs based on methods referenced in Table 7.1. Field measurements obtained during cone penetrometer testing using ultraviolet fluorescence (CPT-UVF) and electrical conductivity (CPT-EC) modules will be conducted according to the SOPs in Appendix B.

The SOPs and analytical methods are summarized in Section 7.0. The quality assurance (QA) and quality control (QC) procedures to ensure the DQOs are achieved are presented in Section 3.0.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Conestoga-Rovers & Associates (CRA), as consultant to the WCP Group (Group), has overall responsibility for Pilot Test activities at the Site. En Chem, Inc. of Madison, Wisconsin (En Chem) will perform the analyses of all groundwater samples collected for off-Site analysis. STRATIGRAPHICS, Inc. of Glen Ellyn, Illinois will conduct the on-Site CPT-EC-UVF testing. All firms will provide project management as appropriate to their responsibilities. CRA will provide administrative oversight and QA/QC for all deliverables. CRA will maintain a file copy of all laboratory deliverables. All final project deliverables will be issued by CRA.

A project organization chart showing the relationship and lines of communication among all project participants with QA/QC responsibilities is presented as Figure 2.1. The QA/QC responsibilities of key project personnel are summarized below.

Alan Van Norman - Project Manager - CRA

- general overview of the project to ensure that the WCP Group's objectives are met;
- liaison with U.S. EPA and IEPA;
- data assessment;
- preparation and review of reports; and
- provide technical representation of project activities.

Steven Day - QA/QC Officer - CRA

- perform laboratory system audits;
- overview and review field QA/QC;
- review laboratory QA/QC;
- oversee data validation and assessment;
- advise on data corrective action procedures;
- preparation and review of reports; and
- provide QA/QC representation of project activities.

Steven Wanner - Field QA Officer - CRA

- management of field activities and field QA/QC;

- data assessment;
- technical representation of field activities;
- preparation of SOPs for field activities;
- preparation of reports;
- evidence file custodian; and
- advise on field corrective action procedures.

Kevin Hinckley - Project Manager - En Chem

- ensures all resources of the laboratory are available on an as-required basis;
- overview of final analytical reports; and
- approve final analytical reports prior to submission to CRA.

Glen Coder - Operations Manager - En Chem

- coordinate laboratory analyses;
- supervise in-house chain-of-custody procedures;
- schedule sample analyses;
- oversee data review; and
- oversee preparation of analytical reports.

Gregory Graf - Laboratory QA Officer - En Chem

- overview laboratory quality assurance;
- overview QA/QC documentation;
- conduct detailed data review;
- decide laboratory corrective actions, if required;
- provide technical representation of laboratory QA procedures; and
- coordinate preparation of laboratory SOPs.

Renee Breed - Sample Custodian - En Chem

- receive and inspect the incoming sample containers;
- record the condition of the incoming sample containers;
- sign appropriate documents;
- verify chain-of-custody and its correctness;

- notify appropriate laboratory personnel of sample receipt and inspection;
- initiate transfer of the samples to appropriate lab sections; and
- control and monitor access/storage of samples and extracts.

Primary responsibility for project quality rests with CRA's QA/QC Officer and Field QA Officer. Ultimate responsibility for project quality rests with CRA's Project Manager. Independent quality assurance will be provided by En Chem's Project Manager and QA Officer prior to release of all data to CRA.

U.S. EPA RESPONSIBILITIES

The U.S. Environmental Protection Agency (EPA) Region 5 Remedial Project Manager (RPM) will be responsible for the overview of this project. The RPM will also be responsible for providing approval of the QAPP. Mr. Kevin Adler is the RPM for the Pilot Project activities.

The U.S. EPA Region 5 Quality Assurance Reviewer is responsible for reviewing and for providing final approval of the QAPP. In addition, U.S. EPA Region 5 is responsible for conducting external performance and system audits of laboratory and field activities.

ILLINOIS EPA RESPONSIBILITIES

The Illinois EPA (IEPA) Project Manager will be responsible for oversight of the project for the State of Illinois. Oversight responsibilities include reviewing and commenting on project documents, and providing state rules, regulations, and guidance. Mr. Jerry Willman is the IEPA Project Manager for the Pilot Project activities.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results that are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance, and corrective action are described in subsequent sections of this QAPP.

3.1 PRECISION

3.1.1 DEFINITION

Precision is a measure of the degree to which two or more measurements are in agreement.

3.1.2 FIELD PRECISION OBJECTIVES

Field precision for measurements associated with groundwater monitoring will be assessed through the collection and measurement of duplicate samples or calibration check solutions at a frequency of one per ten groundwater samples. The precision control limits for field measurements obtained during the Pilot Project activities are summarized in the SOPs in Appendix B.

3.1.3 LABORATORY PRECISION OBJECTIVES

Precision in the laboratory will be assessed through the calculation of relative percent differences (RPDs) for replicate/duplicate samples. The equation to be used for calculating precision for this project can be found in Section 12.2 of this QAPP. Precision control limits for laboratory parameters are provided in Table 3.1.

3.2 ACCURACY

3.2.1 DEFINITION

Accuracy is the degree of agreement between an observed value and an accepted reference value.

3.2.2 FIELD ACCURACY OBJECTIVES

Accuracy in the field is assessed through the use of field and trip blank samples and is ensured by observing all sample handling procedures, preservation requirements, and holding time periods. Accuracy of field measurements associated with groundwater monitoring will be assessed by analyzing calibration check solutions. Accuracy control limits for the field measurements obtained during the Pilot Project activities are presented in the SOPs in Appendix B.

3.2.3 LABORATORY ACCURACY OBJECTIVES

Laboratory accuracy will be assessed by determining percent recoveries from the analysis of matrix spikes (MS), laboratory control samples (LCS), or standard reference materials (SRMs). The equation to be used for calculating accuracy for this project can be found in Section 12.1 of this QAPP. Accuracy control limits for laboratory parameters are provided in Table 3.2.

Analytical sensitivity is also a measure of laboratory accuracy. The sensitivities required for the analyses will be the targeted quantitation limits presented in Table 3.3.

3.3 COMPLETENESS

3.3.1 DEFINITION

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.3.2 FIELD COMPLETENESS OBJECTIVES

Field completeness is a measure of the amount of valid field measurements obtained from all the measurements taken during the project. The equation for completeness is presented in Section 12.3 of this QAPP. The field completeness objective for this project will be greater than 90 percent.

3.3.3 LABORATORY COMPLETENESS OBJECTIVES

Laboratory completeness is a measure of the amount of valid laboratory measurements obtained from all the measurements taken during the project. The equation for completeness is presented in Section 12.3 of this QAPP. The laboratory completeness objective for this project will be greater than 95 percent.

3.4 REPRESENTATIVENESS

3.4.1 DEFINITION

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

3.4.2 MEASURES TO ENSURE REPRESENTATIVENESS OF FIELD DATA

Representativeness is dependent upon the proper design of the sampling program. Representativeness will be ensured by following the proper sampling protocols and using proper sampling techniques.

3.4.3 MEASURES TO ENSURE REPRESENTATIVENESS OF LABORATORY DATA

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing field duplicate samples. The sampling network is designed to provide data representative of Site conditions. During development of this network, consideration has been given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the Superfund program. The rationale for the sampling network is discussed in Section 5.0 of the Work Plan provided in Appendix A.

3.5 COMPARABILITY

3.5.1 DEFINITION

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

3.5.2 MEASURES TO ENSURE COMPARABILITY OF FIELD DATA

Comparability is dependent upon the proper design of the sampling program and will be ensured by using proper sampling techniques.

3.5.3 MEASURES TO ENSURE COMPARABILITY OF LABORATORY DATA

The laboratory data to be obtained during the Pilot Project activities will be comparable to previous data when similar sampling and analytical methods are used. Comparability is also dependent on similar QA objectives.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Trip blank, method blank, field duplicate, matrix spike, LCS, and laboratory duplicate samples will be analyzed to assess the quality of the laboratory's data resulting from the field sampling and analysis program. Field blank samples will not be collected for the pilot project as all sampling equipment will be dedicated and/or pre-cleaned prior to use.

Trip blank samples are used to assess the potential for contamination of samples resulting from contaminant migration during sample shipment and storage. Trip blank samples pertain only to aqueous VOC samples. Trip blank samples that consist of ultra pure water are prepared in sample containers at the laboratory prior to the sampling event and are kept with the groundwater samples collected from extraction wells throughout the sampling event. Trip blank samples will be packaged for shipment with other groundwater samples and submitted to the laboratory for analysis. One trip blank sample will be included in each cooler used to ship groundwater samples collected for VOC analysis. Trip blank sample containers will not be opened prior to analysis at the laboratory.

Method blank samples are generated within the laboratory and are used to assess contamination resulting from laboratory procedures.

Field duplicate samples are analyzed to assess the overall sampling and analytical reproducibility. Field duplicate samples are collected by alternately filling the sample containers for each parameter to be analyzed from the same sampling device. Field duplicate samples will be collected only for samples collected before startup and after shutdown of each pilot treatment system.

Matrix spikes provide information about the effect of the sample matrix on the preparation and measurement methodology. Matrix spike samples generally are analyzed in duplicate and are referred to as matrix spike/matrix spike duplicate (MS/MSD) samples. MS/MSD samples are investigative samples which have been fortified (spiked) by the laboratory with a known amount of the analyte(s) of interest. Aqueous MS/MSD samples must be collected at triple the usual volume for VOCs, and double the usual volume for extractable organics (e.g., semivolatile organic compounds). *Site-specific MS/MSD samples will be designated only for samples* collected before startup and after shutdown of each pilot treatment system.

The level of the QC effort for samples collected before startup and after shutdown of each pilot treatment system will be one field duplicate sample for every ten or fewer samples. One VOC trip blank sample consisting of laboratory-prepared ultra pure water will be included along with each shipment of groundwater VOC samples. One MS/MSD sample will be submitted with every 20 or fewer samples collected before startup and after shutdown of each pilot treatment system for organic, inorganic, or general chemistry analyses. Alternately, matrix spike/laboratory duplicate (MS/DUP), laboratory control sample/laboratory duplicate (LCS/DUP), or duplicate laboratory control samples (LCS/LCD) may be designated for some general chemistry analyses. The SOPs in Appendix B detail whether MS/MSD, MS/DUP, LCS/DUP, or LCS/LCD samples are analyzed for a particular method.

The level of QC effort for samples associated with samples collected during pilot testing and the bromide tracer testing will consist of the analysis of internal laboratory QC samples at the frequencies specified in the SOPs in Appendix B. Site-specific QC samples will not be collected or designated for these samples as the data from these tests will be used for engineering evaluation of the treatment units during the testing period and to characterize the extracted water. Therefore, a reduced level of QC effort is appropriate for these tests. However, the laboratory will use samples from the pilot tests as matrix spike and laboratory duplicate samples if sufficient samples are submitted to comprise a batch of samples. Samples collected to determine contaminant mass removal (i.e., data from samples collected before startup and after shutdown of the pilot treatment systems) will require the level of QC effort detailed in Table 1.1.

The level of QC effort for the field measurement of pH consists of daily calibration and periodic calibration verification using two standard reference solutions as appropriate to the sample pH. The level of QC effort for field conductivity, oxidation-reduction potential (ORP), dissolved oxygen (DO), and turbidity measurements will include periodic calibration verification of the instrument using standard solutions of known conductivity, ORP, DO, and turbidity. Temperature measurements are obtained with pH and/or conductivity and field calibration is neither possible nor practical. The field measurement of residual chlorine concentrations in tap water will be performed using test strips (EM Quant or equivalent). No calibration or external QC analyses are required for this test.

The level of QC effort for the CPT-EC module will consist of checking the electrical conductivity channel output using a series of calibration resistors. The level of QC effort for the CPT-UVF module will consist of checking the photonic sensor operation by exposing the sensor to ambient light (high light sensor), by covering the sensor (dark signal), and by exposing a fluorescent material to the UV light source and monitoring the photonic sensor response. Additional details for the CPT-EC-UVF protocols are provided in the SOPs in Appendix B.

The number of QC samples to be collected for the Pilot Project activities is provided in Table 1.1.

4.0 SAMPLING PROCEDURES

The sampling procedures for the various media at the Site are provided in the Pilot Project Sampling and Analysis Plan. Table 4.1 details the sample preservation, sample container, sample holding time, sample packaging, and sample shipping requirements for the analyses to be performed on the samples collected during the Pilot Project activities. En Chem will provide pick-up and delivery service for the project. However, if it is necessary to ship samples, they will be shipped to:

En Chem, Inc.
525 Science Drive
Madison, Wisconsin 53711
Attn.: Sample Receiving
Phone: 608-232-3312 (888-536-2436)
Fax: 608-233-0502

Sample containers will be precleaned by En Chem, or purchased precleaned, as described in the U.S. EPA guidance document entitled "Specifications and Guidance for Contaminant-Free Sample Containers", EPA 540/R-93/051.

5.0 SAMPLE CUSTODY AND DOCUMENT CONTROL

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for evidence admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all original laboratory reports, are maintained under document control in a secure area.

A sample or evidence file is in a person's custody if:

- the item is in actual possession of a person; or
- the item is in the view of the person after being in actual possession of the person; or
- the item was in actual physical possession but is locked up to prevent tampering; or
- the item is in a designated and identified secure area.

5.1 FIELD CHAIN-OF-CUSTODY PROTOCOLS

Field logbooks will provide the means of recording the data collecting activities performed. As such, logbook entries will be described in as much detail as possible so that persons going to the Site could reconstruct a particular situation solely from the logbook entries.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel and will be stored in the on-Site treatment building when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- person to whom the logbook is assigned;

- logbook number;
- project name;
- project start date; and
- end date.

Entries into the logbook will contain a variety of information. At the beginning of each day's logbook entry, the date, start time, weather, names of all sampling team members present, and the signature of the person making the entry will be entered. The names of individuals visiting the Site or field sampling team and the purpose of their visit will also be recorded in the field logbook.

All field measurements taken and samples collected will be recorded. All logbook entries will be made in ink, signed and dated with no erasures. If an incorrect logbook entry is made, the incorrect information will be crossed out with a single strike mark, which is initialed and dated by the person making the erroneous entry. The correct information will be entered into the logbook adjacent to the original entry.

Whenever a sample is collected or a measurement is made, a detailed description of the location will be recorded in the logbook. Photographs taken at a location, if any, will also be noted in the logbook. All equipment used to obtain field measurements will be recorded in the field logbook. In addition, the calibration data for all field measurement equipment will be recorded in the field logbook.

CPT-EC-UVF measurements will be recorded by STRATIGRAPHICS using log sheets and data logging software.

Samples will be collected following the sampling procedures documented in the Pilot Project SAP. The equipment used to collect samples, time of sample collection, sample description, depth from which soil samples were collected, and volume and number of containers will be recorded in the field logbook.

Site-specific sample identification numbers will be assigned to each sample when it is collected. The Site-specific sample numbering system is provided in the SAP. Field blank and field duplicate samples will be submitted blindly to the laboratory using this numbering system. Trip blank samples and samples selected for MS/MSD or LCS/DUP analysis will not be submitted blindly to the laboratory.

The sample packaging and shipping procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred to another person or the laboratory. As few people as possible will handle the samples.

2. All sample containers will be identified by using sample labels which include the date of collection and analyses to be performed.

3. Sample labels will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook entry would explain that a pencil was used to fill out the sample label because the ball-point pen would not function in freezing weather.

4. Samples will be accompanied by a properly completed chain-of-custody form. The sample identification numbers will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign and record the date and time on the form. The chain-of-custody form documents sample custody transfers from the sampler to another person, to the laboratory, or to/from a secure storage area. A typical chain-of-custody form is presented on Figure 5.1

5. Samples will be properly packaged for shipment (see Table 4.1) and dispatched to En Chem for analysis, with a separate signed chain-of-custody form enclosed in and secured to the inside top of each sample cooler. Shipping coolers will be secured with custody tape for shipment to the laboratory. The custody tape is then covered with clear plastic tape to prevent accidental damage to the custody tape.

6. Whenever samples are collocated with a government agency, a separate chain-of-custody form will be prepared for those samples and marked to indicate with whom the samples are being collocated. The person relinquishing the samples to the agency should request the representative's signature

acknowledging sample receipt. If the representative is unavailable or refuses to sign, this is noted in the "Received By" space on the chain-of-custody form.

7. All sample shipments will be accompanied by the chain-of-custody form identifying its contents. The chain-of-custody form is a four part carbonless-copy form. The form is completed by the sampling team that, after signing and relinquishing custody to the shipper, retains the bottom (goldenrod) copy. The shipper, if different than the sampling team members, retains the pink copy after relinquishing custody to the laboratory. The yellow copy is retained by the laboratory and the fully executed white copy is returned as part of the data deliverables package.

8. If the samples are sent by common carrier, a bill of lading will be used and copies will be retained as permanent documentation. Commercial carriers are not required to sign the chain-of-custody form as long as the form is sealed inside the sample cooler and the custody tape remains intact.

9. Samples will usually be transported or shipped to the laboratory the same day the samples are collected in the field.

5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

Laboratory custody and document control procedures will be carried out as specified in the appropriate SOPs in Appendix B.

5.3 STORAGE OF SAMPLES

Following laboratory sample receipt and log-in, all samples will be stored in the appropriate locations. All samples will be stored within an access-controlled location and will be maintained properly preserved until completion of all analytical work or, as a minimum, for 30 days after receipt of the final report by CRA.

5.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

Evidentiary files for the entire project will be maintained by CRA's Field QA Officer and will consist of the following:

- project plan;
- project log books;
- field data records;
- sample identification documents;
- chain-of-custody records;
- correspondence;
- references and literature;
- laboratory data deliverables;
- data validation reports;
- interim project/progress reports;
- QA reports;
- miscellaneous (photos, maps, drawings, etc.); and
- final report.

The evidentiary file materials will be the responsibility of the evidentiary file custodian with respect to file maintenance and document removal.

The laboratory will be responsible for maintaining analytical logbooks and data. Raw laboratory data files will be maintained by the laboratory for a period of five years.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

The procedures for maintaining the accuracy for all the instruments and measuring equipment that will be used for conducting field tests and laboratory analyses are described below. The instruments and equipment used for sample analysis and field measurements will be calibrated prior to each use or on a scheduled, periodic basis.

6.1 FIELD INSTRUMENTS/EQUIPMENT

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of the data are consistent with the manufacturer's specification and the SOPs in Appendix B. Routine equipment used for field measurement will include a pH/temperature meter, turbidity meter, DO meter, ORP meter, and conductivity meter. The meters to be used are specified in the field SOPs or will be equipment with equivalent capability.

Equipment to be used during field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed to ensure that any prior equipment problems are not overlooked and all necessary repairs to equipment have been completed.

6.1.1 FIELD INSTRUMENT CALIBRATION

Field equipment will be calibrated, operated, and maintained in a manner consistent with the manufacturer's guidelines and the field SOPs in Appendix B.

6.2 LABORATORY INSTRUMENTS

Calibration of laboratory equipment will be based on approved written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QA activities. These records will be filed at the location where the work is performed and will be subject to QA audit. The laboratory will maintain a properly trained repair staff with in-house spare parts for all instruments, or will maintain instrument service contracts.

The records of calibration will be kept as follows:

- Each instrument's calibration history will be maintained in logbooks kept with each instrument;

- Records for each instrument, which includes manufacturer, model numbers, date of last calibration and by whom calibrated (signature), due date of next calibration, and compensation or correction figures, are maintained by the laboratory QA Officer;

- A written stepwise calibration procedure will be available for each instrument; and

- Any instrument that is not calibrated to with the manufacturer's original specification will display an appropriate warning tag or otherwise will be removed from service.

Specific calibration procedures are detailed in the appropriate SOPs presented in Appendix B.

7.0 ANALYTICAL PROCEDURES

The samples collected for chemical analyses will be analyzed using the methods listed in Table 7.1 and are detailed in the respective SOPs included in Appendix B. Field measurements associated with groundwater monitoring will be obtained using the methods in Table 7.1 and are detailed in the SOPs in Appendix B. Groundwater level measurements will be obtained using an electric water level tape. CPT-EC-UVF measurements will be obtained using the procedures described in the STRATIGRAPHICS SOPs in Appendix B.

8.0 INTERNAL QUALITY CONTROL AND FREQUENCY

This section presents the internal quality control checks that will be employed for field and laboratory measurements and the frequencies at which they will be performed.

8.1 FIELD QC

Quality control procedures for field measurements will be limited to checking the reproducibility of the measurement in the field by obtaining multiple readings and by calibrating the instruments (where appropriate).

Quality control for samples collected before startup and after shutdown of each pilot treatment system will involve collecting field duplicates and trip blanks in accordance with the applicable procedures and frequencies described in Section 3.0, and the level of effort indicated in Table 1.1.

8.2 LABORATORY QC

Specific procedures related to internal laboratory QC samples are detailed in the following subsections. The internal QC checks will be consistent with the requirements of the methods specified in Table 7.1. The evaluation criteria outlined in the SOPs presented in Appendix B will be used by laboratory personnel to assess the QC data.

8.2.1 INSTRUMENT CALIBRATION

The compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. The initial calibration demonstrates that the instrument is capable of acceptable performance prior to sample analysis. Continuing calibration checks document that the initial calibration is still valid, and that satisfactory maintenance and adjustment of the instrument is achieved on a day-to-day basis. The specific control criteria and corrective action requirements for initial and continuing calibrations will be as specified in the appropriate SOPs presented in Appendix B.

8.2.2 INSTRUMENT PERFORMANCE CHECKS

Performance checks ensure mass resolution, identification, and sensitivity of the gas chromatography/mass spectrometry (GC/MS) instruments. Prior to calibration, each instrument must be successfully "tuned" using the performance check standards specified in the methods. The acceptance criteria are detailed in the appropriate SOPs in Appendix B.

8.2.3 INTERNAL STANDARDS PERFORMANCE

Internal standards percent recovery and retention time evaluations ensure that GC/MS sensitivity and response is stable during every analysis. Acceptance criteria are specified in the appropriate SOPs presented in Appendix B.

8.2.4 SURROGATE COMPOUNDS

Surrogate compounds are used to monitor overall method performance for the organic analyses. Every blank, standard and sample (including MS/MSD samples) will be fortified with surrogate compounds prior to sample preparation. Surrogates will be spiked into samples as specified in the SOPs in Appendix B. Surrogate spike percent recoveries are required to be within the control limits specified in the method for samples not requiring dilutions. Dilution of samples to bring the detected analyte concentrations into the instrument's calibration range may dilute the surrogates below the quantitation range. Assessment of data quality in these cases will be based on the quality control data from the QC check, matrix spike, and matrix spike duplicate samples. The surrogate spike percent recovery acceptance criteria are specified in Table 8.1.

8.2.5 BLANK SAMPLES

Blank samples, including preparation or method blanks and instrument blanks, will be analyzed at the required frequencies specified in the SOPs. Preparation or method blank samples, consisting of an aliquot of analyte-free water, will be carried through the entire analytical procedure to determine the existence and magnitude of contamination resulting from laboratory activities. Instrument blanks consist of analyte-free water or solvent used to determine whether or not the instrument is free of contaminants and ready to calibrate and analyze samples. The specific acceptance criteria and corrective action requirements are specified in the appropriate SOPs presented in Appendix B.

8.2.6 MS/MSD, MS/DUP, LCS/DUP, AND LCS/LCD SAMPLES

A MS/MSD sample set will be analyzed by the laboratory at a minimum frequency of 1 per 20 or fewer samples for organic parameters. A MS/MSD or MS/DUP sample set will be analyzed by the laboratory at a minimum frequency of 1 per 20 or fewer samples for inorganic parameters. Percent spike recoveries will be used to evaluate analytical accuracy relative to the sample matrices. The RPD of the MS/MSD or laboratory duplicate data will be used to assess analytical precision relative to the sample matrices. The percent recovery and RPD acceptance criteria are detailed in Tables 3.1 and 3.2.

The MS data for metals analysis is used to provide information about the effect of each sample matrix on the sample preparation procedures and measurement methodology. The spike may be added before digestion (pre-digestion spike) or after completion of digestion procedures (post-digestion spike). The percent recovery acceptance criteria for pre-digestion spikes are detailed in Table 3.2.

A LCS/laboratory duplicate sample set will be analyzed for total suspended solids. LCSs consist of standard reference materials added to purified water and are analyzed to check the accuracy of the analytical methods independent of sample matrix interferences. Laboratory duplicate sample data will be used to assess the precision of the analytical methods.

LCS/LCD samples will be analyzed if insufficient volume is available for MS/MSD or MS/DUP analysis. Percent recovery data will be used to monitor analytical accuracy and RPD will be used to monitor analytical precision.

8.2.7 INTERFERENCE CHECK SAMPLES

Interference check samples (ICS) are used to verify that proper inter-element correction factors have been established for analyses performed using inductively coupled plasma (ICP) spectroscopy methods. The ICP ICS is analyzed before and after each batch of samples. The acceptance criteria are detailed in the appropriate SOPs in Appendix B.

8.2.8 ICP SERIAL DILUTION SAMPLES

ICP serial dilution samples are analyzed to determine whether significant chemical or physical interferences exist due to the sample matrix. The acceptance criteria are detailed in the appropriate SOP in Appendix B.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

The laboratory will perform analytical data reduction, review, and reporting under the direction of the laboratory QA Officer who will be responsible for assessing data quality and advising of any data that were rated "preliminary" or "unacceptable" or other qualifications based on the established QC acceptance criteria. Data reduction, review, and reporting typically will be conducted as detailed in the following:

1. Raw data produced and checked by the responsible analyst are turned over for independent review by another analyst.

2. The area supervisor or his designee reviews the data for attainment of QC acceptance criteria established by this QAPP.

3. The area supervisor or his designee will decide whether any sample re-analysis is required.

4. Upon completion of all reviews and acceptance of the raw data by the supervisor, a report will be generated and sent to the laboratory Project Manager.

5. The laboratory Project Manager will complete a thorough inspection of all reports.

6. Upon acceptance of the preliminary reports by the laboratory Project Manager, final reports will be generated and signed by the laboratory Project Manager.

7. Routine audits of selected data packages are performed by the laboratory Quality Assurance Officer or his/her designee.

Laboratory data reduction will be performed using the equations in the appropriate SOPs provided in Appendix B. Field measurement data from direct-reading instruments will not require reduction. Reduction of CPT-EC-UVF data will be performed by STRATIGRAPHICS.

Validation of the laboratory data will be performed by CRA's QA/QC Officer or his designee based on the relevant and applicable evaluation criteria outlined in "National Functional Guidelines for Organic Data Review", EPA-540/R-99/008, October 1999 and "National Functional Guidelines for Inorganic Data Review", EPA-540/R-94-013, February 1994. The assessment of laboratory data will include checks for adherence to the laboratory QA procedures and the accuracy and precision acceptance criteria presented in this QAPP, the presence of transcription errors, and anomalously high or low parameter values. The results of the data validation procedure will be summarized in a memorandum and reported to CRA's Project Manager. The data validation memoranda will detail any violations of QC acceptance criteria and their effects upon the usability of the data.

Field data and sample collection activities that are presented in project reports will be appropriately identified and appended to the report. Where data have been reduced or summarized, the method of reduction will be documented in the report. In addition, field data will be audited for anomalously high or low values that may appear to be inconsistent with other Site data.

Data packages for the laboratory analyses will consist of the following deliverables:

- a case narrative that includes a summary of analytical methods used and a description of any unusual action or conditions;
- dates of sample receipt, extraction/digestion, and analysis;
- laboratory and field sample identification numbers;
- samples results in tabular format;
- method blank sample data summaries;
- surrogate compound recovery data and control limits;
- MS/MSD, MS/DUP, LCS/DUP, LCS/LCD percent recovery, RPD data, and control limits;

- QC check sample data and control limits; and
- executed chain-of-custody forms.

The data packages will be stored with the evidentiary files as described in Section 5.4.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in this QAPP. The audits of field and laboratory activities include two separate, independent parts: internal and external audits.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 INTERNAL FIELD AUDITS

10.1.1.1 INTERNAL FIELD AUDIT RESPONSIBILITIES

An internal audit of field activities (sampling and measurements) will be conducted by CRA's Field QA Officer or his designee.

10.1.1.2 INTERNAL FIELD AUDIT FREQUENCY

Field audits will verify that the procedures established by this QAPP and the Pilot Project SAP are being followed. These audits will be conducted at project start-up to identify deficiencies in the field sampling and documentation procedures. Any deficiencies identified will be documented and corrective actions will be taken to rectify the deficiencies. Follow-up audits will be performed as necessary to verify that deficiencies have been corrected, and that QA procedures are maintained throughout the project.

10.1.1.3 INTERNAL FIELD AUDIT PROCEDURES

Internal field audits will include examination of field sampling records, chain-of-custody documentation, field instrument operating records, and field instrument calibration records. In addition, sample collection, handling and packaging procedures will be reviewed during field audits.

10.1.2 EXTERNAL FIELD AUDITS

10.1.2.1 EXTERNAL FIELD AUDIT RESPONSIBILITIES

External field audits may be conducted by U.S. EPA Region 5.

10.1.2.2 EXTERNAL FIELD AUDIT FREQUENCY

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the U.S. EPA Region 5.

10.1.2.3 OVERVIEW OF THE EXTERNAL FIELD AUDIT PROCESS

External field audits may be conducted according to the field activity information presented in the QAPP.

10.2 LABORATORY SYSTEMS AND PERFORMANCE AUDITS

10.2.1 INTERNAL LABORATORY AUDITS

10.2.1.1 INTERNAL LABORATORY AUDIT RESPONSIBILITIES

Internal laboratory audits will be conducted by the laboratory's QA Officer.

10.2.1.2 INTERNAL LABORATORY AUDIT FREQUENCY

Internal system audits of the laboratory will be conducted on an annual basis and internal performance audits will be conducted on a quarterly basis.

10.2.1.3 INTERNAL LABORATORY AUDIT PROCEDURES

Systems audits will include examining laboratory documentation of sample receipt and log-in, sample storage, chain-of-custody procedures, sample preparation and analysis procedures, and instrument operating and QC records.

Performance audits will consist of the laboratory's QA Officer preparing and submitting blind QC samples to the laboratory for analysis. The laboratory QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QA performance.

10.2.2 EXTERNAL LABORATORY AUDITS

10.2.2.1 EXTERNAL LABORATORY AUDIT RESPONSIBILITIES

An external audit may be conducted by U.S. EPA Region 5.

10.2.2.2 EXTERNAL LABORATORY AUDIT FREQUENCY

An external laboratory audit may be conducted at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of the U.S. EPA.

10.2.2.3 OVERVIEW OF THE EXTERNAL LABORATORY AUDIT PROCESS

External laboratory audits will include, but not be limited to, review of laboratory analytical procedures, laboratory on-site audits, and/or submitting performance evaluation samples to the laboratory for analysis.

11.0 PREVENTIVE MAINTENANCE

All analytical instruments to be used for this project will be serviced by the laboratory personnel at regularly scheduled intervals in accordance with the manufacturer's recommendations. Instruments may also be serviced at other times due to failure. Requisite servicing beyond the abilities of the laboratory personnel will be performed by the equipment manufacturer or its designated representative.

Daily checks of each instrument will be conducted by the analyst who has been assigned responsibility for that instrument. These checks will include changing GC inlet liners, tuning GC/MS instruments, checking operation of data systems, and checking for leaks. Manufacturer's recommended procedures will be followed in every case.

Table 11.1 presents routine preventive maintenance for laboratory and field instruments associated with groundwater and tap water monitoring. Additional routine preventive maintenance information is contained in the SOPs in Appendix B. Routine preventive maintenance information for the CPT-EC-UVF equipment is provided in the SOPs in Appendix B.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

The following sections include the procedures and formulae utilized to assess the levels of precision, accuracy, and completeness achieved during the associated sample analyses.

12.1 ACCURACY ASSESSMENT

In order to ensure the accuracy of the analytical procedures, an environmental sample will be designated by the sampler, or a sample will be randomly selected by the laboratory, and spiked with a known amount of the analyte or analytes to be evaluated. In general, a sample spike will be included in and analyzed with every batch of 20 samples analyzed on each instrument. The analyte concentration in the spiked sample compared to the analyte concentration in the unspiked sample will be used to determine percent recovery. The percent recovery (%R) for a spiked sample is calculated according to the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \times 100$$

Percent recovery control charts for each spiked analyte will be maintained on a matrix-specific basis.

12.2 PRECISION ASSESSMENT

MS/MSD samples are prepared by choosing a designated sample or a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and spiking each of the aliquots with a known amount of analyte. The duplicate spiked sample is then included in the analytical sample batch. The analysis of MS/MSD samples provides information regarding the precision of the preparation and analytical techniques. The RPD of the MS and MSD will be calculated and plotted on control charts. The RPD is calculated using the following formula:

$$\text{RPD} = \frac{|\text{Amount in Spike 1} - \text{Amount in Spike 2}|}{0.5(\text{Amount in Spike 1} + \text{Amount in Spike 2})} \times 100$$

The RPDs for laboratory duplicates and duplicate laboratory control samples are determined similarly, but the samples are not spiked prior to analysis.

12.3 COMPLETENESS ASSESSMENT

Completeness is the number of valid sample results compared to the total number of sample results of a specific matrix analyzed using a specific method. Following completion of the analytical testing, the percent completeness will be calculated using the following equation:

$$\text{Completeness} = \frac{(\text{Number of Valid Measurements})}{(\text{Number of Measurements Planned})} \times 100$$

13.0 CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving, and implementing measures to rectify unacceptable procedures or out-of-control QC performance that can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All corrective actions proposed and implemented will be documented.

13.1 FIELD CORRECTIVE ACTION

Corrective action in the field may be necessary when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP) and when sampling procedures and/or field analytical procedures require modification due to unexpected conditions. U.S. EPA will be notified of any field changes. In general, the field sampling team may identify the need for corrective action. The field sampling team, in consultation with the Field QA Officer, will recommend a corrective action. The Field QA Officer will approve the corrective action, which will be implemented by the field team. It will be the responsibility of the Field QA Officer to ensure the corrective action has been implemented.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to the use of unapproved methods or improper use of approved methods. The Field QA Officer will identify deficiencies and recommend corrective action to CRA's Project Manager. Implementation of corrective actions will be performed by the Field QA Officer. Corrective action will be documented in quality assurance reports to management.

13.2 LABORATORY CORRECTIVE ACTION

Corrective action in the laboratory may occur prior to, during, or after the initial sample analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, or potentially high concentration samples may be identified during sample log-in or prior to analysis. Following consultation with analysts and area supervisors, it may be necessary for the laboratory QA Officer to approve the implementation of corrective action. The submitted SOPs specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup or automatic re-injection/re-analysis when certain QC criteria are not met. The SOPs in Appendix B each provide a discussion on corrective action requirements.

The analyst will identify the need for corrective action. The Operations Manager or area supervisors, in consultation with the laboratory staff, will approve the required corrective action to be implemented by the laboratory staff. The laboratory QA Officer will ensure implementation and documentation of the corrective action.

All laboratory corrective actions will be completed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action report and the case narrative report sent with the laboratory report.

The need for corrective action may also be identified during systems or performance audits. In these cases, the need for corrective action will be identified by the auditor and the corrective action taken to resolve the problem will be documented by the laboratory QA Officer. The corrective action taken will depend upon the QA/QC criteria which was violated. All problems requiring corrective action and the corrective action taken will be reported to the laboratory Project Manager.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

CRA's QA/QC Officer may identify the need for corrective action during either data validation or data assessment. Potential types of corrective action may include resampling by the field team or reanalysis of samples by the laboratory.

The corrective actions taken are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded). Should CRA's QA/QC Officer identify a situation requiring corrective action, CRA's Project Manager will be responsible for approving the implementation of corrective action.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Management (including U.S. EPA and IEPA) will receive a report on the performance of the measurement system and data quality following the completion of the project. This report will be included in Pilot Project Report submitted to U.S. EPA and IEPA.

Minimally, this report will include:

- assessment of measurement data quality indicators (i.e., data accuracy, precision, and completeness);
- any changes in the QA/QC program;
- results of external system audits (if conducted by U.S. EPA and the results provided to CRA); and
- QA problems, corrective actions taken, and resolutions.

CRA's QA/QC Officer will be responsible within the organizational structure for including the QA information in the Pilot Project Report. The QA information will be summarized in a section of the Pilot Project Report that will provide an overall data assessment in accordance with the data quality objectives outlined in this QAPP.

FIGURES

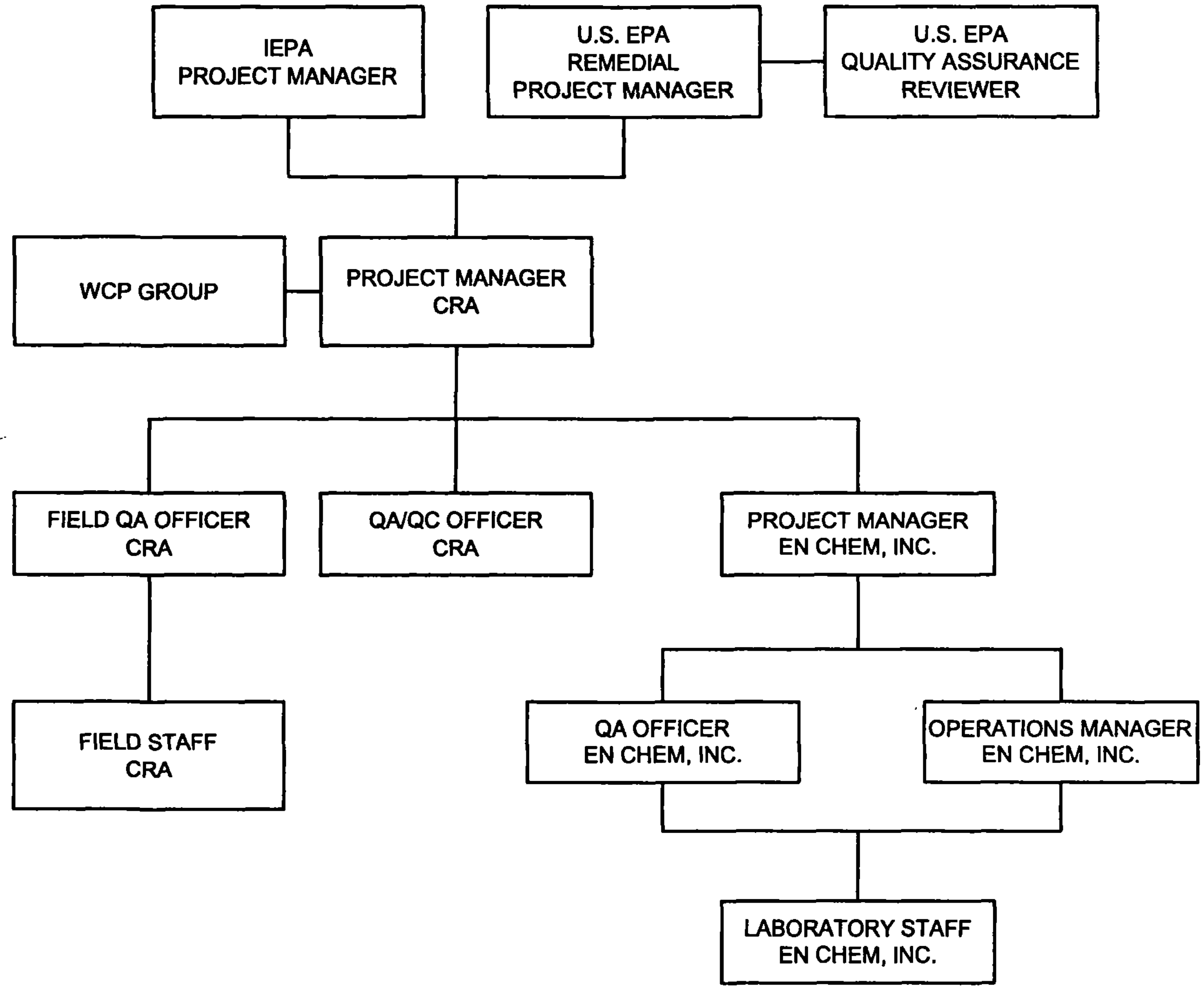

figure 2.1

PROJECT QA/QC ORGANIZATION CHART
WCP SITE PILOT PROJECT
Waukegan, Illinois

CRA

15670-00(003)GN-CO001 OCT 10/2000

CRA
CONESTOGA–ROVERS & ASSOCIATES
8615 West Bryn Mawr Avenue
Chicago, IL 60631 (773)380–9933

CHAIN OF CUSTODY RECORD

SHIPPED TO (Laboratory Name):

REFERENCE NUMBER:

PROJECT NAME:

SAMPLER'S SIGNATURE: ______ PRINTED NAME: ______

SEQ. No.	DATE	TIME	SAMPLE No.	SAMPLE MATRIX	No. OF CONTAINERS	PARAMETERS										REMARKS
			TOTAL NUMBER OF CONTAINERS													

SAMPLE

RELINQUISHED BY: ①	DATE: TIME:	RECEIVED BY: ②	DATE: TIME:
RELINQUISHED BY: ②	DATE: TIME:	RECEIVED BY: ③	DATE: TIME:
RELINQUISHED BY: ③	DATE: TIME:	RECEIVED BY: ④	DATE: TIME:

METHOD OF SHIPMENT: AIR BILL No.

White –Fully Executed Copy Yellow –Receiving Laboratory Copy Pink –Shipper Copy Goldenrod –Sampler Copy	SAMPLE TEAM:	RECEIVED FOR LABORATORY BY: DATE:______ TIME:______

figure 5.1

TYPICAL CHAIN–OF–CUSTODY RECORD
WCP SITE PILOT PROJECT
Waukegan, Illinois

CRA

15670–00(003)GN–C0002 JUN 09/2000

TABLES

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Pilot Cell	*Purpose*	*Week Number*	*Sampling Frequency*	*Locations Sampled*	*Field Parameters*	*Laboratory Parameters*[2]	*Number of Samples*	*QC Samples*[1] *Field Duplicates*	*MS/MSD*[3] *MS/DUP*	*Total*
E/R Unit	Contaminant Mass Removal Determination	Week 1	Daily	WN-1	ORP, Temperature, Conductivity, pH, DO	Total Phenolics	35	0	0	35
						Arsenic	35	0	0	35
						Ammonia	35	0	0	35
		Weeks 2 through 4	3 Events/ Week	WN-1	ORP, Temperature, Conductivity, pH, DO	Total Phenolics	45	0	0	45
						Arsenic	45	0	0	45
						Ammonia	45	0	0	45
		Weeks 1 through 4	3 Events/ Week	WN-2	ORP, Temperature, Conductivity, pH, DO	Total Phenolics	60	0	0	60
						Arsenic	60	0	0	60
						Ammonia	60	0	0	60
								0	0	
		Weeks 1 through 4	Weekly	WN-1, WN-2	ORP, Temperature, Conductivity, pH, DO	VOCs	40	0	0	40
						SVOCs	40	0	0	40
						Cyanide	40	0	0	40
						Thiocyanate	40	0	0	40
		Week 1	Once Prior to Startup	WN-1, WN-2	ORP, Temperature, Conductivity, pH, DO, Turbidity	Total Phenolics	10	1	1	12
						Arsenic	10	1	1	12
						Ammonia	10	1	1	12
						VOCs	10	1	1	12
						SVOCs	10	1	1	12
						Cyanide	10	1	1	12
						Thiocyanate	10	1	1	12
		One Week and Four Weeks Following Unit Shutdown	Twice	WN-1, WN-2	ORP, Temperature, Conductivity, pH, DO, Turbidity	Total Phenolics	20	2	1	23
						Arsenic	20	2	1	23
						Ammonia	20	2	1	23
						VOCs	20	2	1	23
						SVOCs	20	2	1	23
						Cyanide	20	2	1	23
						Thiocyanate	20	2	1	23

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Pilot Cell	Purpose	Week Number	Sampling Frequency	Locations Sampled	Field Parameters	Laboratory Parameters[2]	Number of Samples	QC Samples[1] Field Duplicates	QC Samples[1] MS/MSD[3] MS/DUP	Total
E/R Unit	Tracer Test	Week 1	Daily	WN-1, EW-1 EW-2, EW-3, RW-2	ORP, Temperature, Conductivity, pH, DO	Bromide	63	0	0	63
		Week 1	Once 2 Hours Before Startup	RW-2	None	Bromide	1	0	0	1
		Weeks 2 through 4	3 Events/ Week	WN-1, EW-1 EW-2, EW-3, RW-2	ORP, Temperature, Conductivity, pH, DO	Bromide	81	0	0	81
	Extracted Water	Weeks 1 through 4	3 Events/ Week	EW-1, EW-2, EW-3	None	Total Phenolics	36	0	0	36
						Arsenic	36	0	0	36
						Ammonia	36	0	0	36
						VOCs	36	0	0	36
						SVOCs	36	0	0	36
						Cyanide	36	0	0	36
						Thiocyanate	36	0	0	36
						Nitrate	36	0	0	36
						COD	36	0	0	36
						Alkalinity	36	0	0	36
						TSS	36	0	0	36
E Unit	Contaminant Mass Removal Determination	Weeks 1, 3, 5, 7	3 Events/ Week	WN-3	ORP, Temperature, Conductivity, pH, DO	Total Phenolics	60	0	0	60
						Arsenic	60	0	0	60
						Ammonia	60	0	0	60
		Weeks 1, 3, 5, 7	Once/Week	WN-3	ORP, Temperature, Conductivity, pH, DO	VOCs	20	0	0	20
						SVOCs	20	0	0	20
						Cyanide	20	0	0	20
						Thiocyanate	20	0	0	20

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Pilot Cell	Purpose	Week Number	Sampling Frequency	Locations Sampled	Field Parameters	Laboratory Parameters[2]	Number of Samples	QC Samples[1] Field Duplicates	QC Samples[1] MS/MSD[3] MS/DUP	Total
E Unit	Contaminant Mass Removal Determination	Week 1	Once Prior to Startup	WN-3	ORP, Temperature, Conductivity, pH, DO, Turbidity	Total Phenolics	5	1	1	7
						Arsenic	5	1	1	7
						VOCs	5	1	1	7
						SVOCs	5	1	1	7
						Cyanide	5	1	1	7
						Thiocyanate	5	1	1	7
		One Week and Four Weeks Following Unit Shutdown	Twice	WN-3	ORP, Temperature, Conductivity, pH, DO, Turbidity	Total Phenolics	10	1	1	12
						Arsenic	10	1	1	12
						VOCs	10	1	1	12
						SVOCs	10	1	1	12
						Cyanide	10	1	1	12
						Thiocyanate	10	1	1	12
	Extracted Water	Weeks 1, 3, 5, 7	3 Events/ Week	EW-4	None	Total Phenolics	12	0	0	12
						Arsenic	12	0	0	12
						Ammonia	12	0	0	12
						VOCs	12	0	0	12
						SVOCs	12	0	0	12
						Cyanide	12	0	0	12
						Thiocyanate	12	0	0	12
						Nitrate	12	0	0	12
						COD	12	0	0	12
						Alkalinity	12	0	0	12
						TSS	12	0	0	12

1 One trip blank sample will be included in each cooler containing multiple groundwater VOC samples

2 Refer to Table 3.3 for specific analytes in each parameter group. Refer to SAP Table 4.1 for location and frequency details.

3 Matrix spike/matrix duplicate (MS/MSD) analyses will be performed for organic analyses. MS/MSD samples will be collected with extra sample volume for water and VOC soil samples. Triple the normal sample volumes will be collected for VOCs and double the normal volumes will be collected for extractable organics MS/MSD or matrix spike/laboratory duplicate (MS/DUP) analyses will be performed for inorganics analyses. Samples designated for MS/MSD or MS/DUP analyses will be collected at a frequency of one per group of twenty (20) or fewer investigative samples.

TABLE 3.2

LABORATORY ACCURACY CONTROL LIMITS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

	% Recovery Control Limits [1]
	Water
Volatile Organic Compounds	
1,1-Dichloroethene	64-134
Trichloroethene	75-120
Benzene	76-131
Toluene	91-119
Chlorobenzene	88-117
Semivolatile Organic Compounds	
1,2,4-Trichlorobenzene	70-104
Acenaphthene	81-101
2,4-Dinitrotoluene	78-102
Pyrene	79-111
N-Nitroso-di-n-propylamine	71-106
1,4-Dichlorobenzene	72-96
Phenol	22-60
2-Chlorophenol	72-96
4-Chloro-3-methylphenol	68-108
4-Nitrophenol	11-78
Pentachlorophenol	63-127
Inorganics	
Arsenic	84-115
Alkalinity	38-142
Ammonia	75-125
Bromide	75-125
Chemical Oxygen Demand	42-150
Cyanide	70-125
Nitrate	75-125
Thiocyanate	75-125
Total Phenolics	75-125
Total Suspended Solids	80-109

[1] Values are the range of percent recoveries allowed for MS/MSD or LCS/LCD sample analyses. Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for validating the data.

TABLE 3.1

LABORATORY PRECISION CONTROL LIMITS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

	% RPD Control Limits [1]
	Water
Volatile Organic Compounds	
1,1-Dichloroethene	14
Trichloroethene	14
Benzene	11
Toluene	13
Chlorobenzene	13
Semivolatile Organic Compounds	
1,2,4-Trichlorobenzene	8
Acenaphthene	6
2,4-Dinitrotoluene	7
Pyrene	10
N-Nitroso-di-n-propylamine	13
1,4-Dichlorobenzene	9
Phenol	12
2-Chlorophenol	10
4-Chloro-3-methylphenol	8
4-Nitrophenol	15
Pentachlorophenol	12
Inorganics	
Arsenic	5
Alkalinity	7
Ammonia	14
Bromide	20
Chemical Oxygen Demand	19
Cyanide	14
Nitrate	20
Thiocyanate	20
Total Phenolics	20
Total Suspended Solids	20

[1] Values are the maximum RPD values allowed for MS/MSD or LCS/LCD sample analyses Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for validating the data.

TABLE 3.2

LABORATORY ACCURACY CONTROL LIMITS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

	% Recovery Control Limits [1]
	Water
Volatile Organic Compounds	
1,1-Dichloroethene	61-145
Trichloroethene	71-120
Benzene	76-127
Toluene	76-125
Chlorobenzene	75-130
Semivolatile Organic Compounds	
1,2,4-Trichlorobenzene	70-104
Acenaphthene	81-101
2,4-Dinitrotoluene	78-102
Pyrene	79-111
N-Nitroso-di-n-propylamine	71-106
1,4-Dichlorobenzene	72-96
Phenol	22-60
2-Chlorophenol	72-96
4-Chloro-3-methylphenol	68-108
4-Nitrophenol	11-78
Pentachlorophenol	63-127
Inorganics	
Arsenic	84-115
Alkalinity	65-137
Ammonia	80-114
Bromide	75-125
Chemical Oxygen Demand	42-150
Cyanide	68-137
Nitrate	75-125
Thiocyanate	75-125
Total Phenolics	75-125
Total Suspended Solids	80-109

[1] Values are the range of percent recoveries allowed for MS/MSD or LCS/LCD sample analyses. Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for validating the data

TABLE 3.3

TARGETED QUANTITATION LIMITS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

SVOCs (µg/L)	*Targeted*
Benzo(a)pyrene	10
Benzo(b)fluoranthene	10
Benzo(g,h,i)perylene	10
Benzo(k)fluoranthene	10
bis(2-Chloroethoxy)methane	10
bis(2-Chloroethyl)ether	10
2,2'-oxybis(1-Chloropropane)	10
bis(2-Ethylhexyl)phthalate	10
Butylbenzylphthalate	10
4-Bromophenylphenyl ether	10
Carbazole	10
4-Chloroaniline	10
2-Chloronaphthalene	10
4-Chlorophenyl phenyl ether	10
Chrysene	10
Dibenz(a,h)anthracene	10
Dibenzofuran	10
1,2-Dichlorobenzene	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
3,3'-Dichlorobenzidine	50
Diethylphthalate	10
Dimethylphthalate	10
Di-n-butyphthalate	10
Di-n-octylphthalate	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Fluoranthene	10
Fluorene	10
Hexachlorobenzene	10
Hexachlorobutadiene	10
Hexachlorocyclopentadiene	10
Hexachloroethane	10
Indeno(1,2,3-cd)pyrene	10
Isophorone	10
2-Methylnaphthalene	10
Naphthalene	10
2-Nitroaniline	25
3-Nitroaniline	25
4-Nitroaniline	25

TABLE 3.3

TARGETED QUANTITATION LIMITS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

	Targeted
SVOCs (µg/L)	
Nitrobenzene	10
N-Nitroso-di-n-propylamine	10
N-Nitrosodiphenylamine (diphenylamine)	10
Phenanthrene	10
Pyrene	10
1,2,4-Trichlorobenzene	10
4-Chloro-3-methylphenol	10
2-Chlorophenol	10
2,4-Dichorophenol	10
2,4-Dimethylphenol	10
2,4-Dinitrophenol	25
4,6-Dinitro-2-methylphenol	25
2-Methylphenol	10
4-Methylphenol	10
2-Nitrophenol	10
4-Nitrophenol	25
Pentachlorophenol	25
Phenol	10
2,4,5-Trichlorophenol	10
2,4,6-Trichlorophenol	10
Inorganics (mg/L)	
Arsenic	0.010
Alkalinity	20
Ammonia	0.1
Bromide	0.2
Chemical Oxygen Demand	10
Cyanide	0.01
Nitrate	0.2
Thiocyanate	0.1
Total Phenolics	0.2
Total Suspended Solids	10

1 Targeted quantitations limits are presented for guidance only and may not be achievable for all samples.

TABLE 4.1

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Matrix	*Parameter* [1]	*Containers* [2]	*Preservatives* [3]	*Maximum Holding Time From Sample Collection* [4]	*Volume of Sample*	*Shipping*	*Packaging*
Groundwater/Process Water							
	VOCs	Three 40-mL glass septum vials	HCl to pH<2 iced	14 days for analysis	Fill completely	Lab or Overnight Courier	Bubble Wrap
	SVOCs	Two 1-liter amber glass bottles	iced	7 days for extraction, 40 days for analysis	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap
	Arsenic	One 500-mL polyethylene bottle	HNO_3 to pH<2 iced	6 months for analysis	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap
	Alkalinity, Bromide, Nitrate, TSS	Two 1-L polyethylene bottles	iced	14 days for Alkalinity; 7 days for TSS 28 days for Bromide 48 hours for Nitrate	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap
	Ammonia, COD	One 1-L polyethylene bottle	H_2SO_4 to pH<2 iced	28 days for analysis	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap
	Thiocyanate	One 500-mL polyethylene bottle	H_2SO_4 to pH<2 iced	14 days for analysis	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap
	Cyanide	One 500-mL polyethylene bottle	NaOH to pH>12	14 days for analysis	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap
	Total Phenolics	One 1-liter amber glass bottle	H_2SO_4 to pH<2 iced	28 days for analysis	Fill to neck of bottle	Lab or Overnight Courier	Bubble Wrap

[1] VOCs - Volatile Organic Compounds
SVOCs - Semivolatile Organic Compounds
TSS - Total Suspended Solids
COD - Chemical Oxygen Demand

[2] To the extent possible, parameters will be combined into as few sample containers as possible with respect to sample preservation requirements.

[3] Samples requiring refrigeration will be shipped with bagged, cubed ice and will be stored by the laboratory at 4°C ± 2°C following sample receipt and log-in.

[4] Sample holding time will be calculated from the time of sample collection to sample analysis.

TABLE 7.1

SUMMARY OF ANALYTICAL METHODS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Parameter[1]	*Preparation Method Reference*[2]	*SOP Number*	*Analysis Method Reference*	*SOP Number*
Groundwater/Process Water				
VOCs	SW-846 5030B	3-VOA-5	SW-846 8260B	3-VOA-5
SVOCs	SW-846 3510C	3-SVO-1	SW-846 8270C	3-SVO-37
Arsenic	SW-846 3010A	MET-3	SW-846 6010B	MET-42
Alkalinity	SM 2320B	WCM-13	SW-846 2320B	WCM-13
Ammonia	EPA 350.1	WCM-25	EPA 350.1	WCM-58
Bromide	EPA 300.0	WCM-60	EPA 300.0	WCM-59/WCM-60
Chemical Oxygen Demand	EPA 410.4	WCM-39	EPA 410.4	WCM-39
Cyanide	EPA 335.4	WCM-37	EPA 335.4	WCM-23
Nitrate	EPA 300.0	WCM-60	EPA 300.0	WCM-59/WCM-60
Thiocyanate	SM 4500-CN-M	WCM-65	SM 4500-CN-M	WCM-65
Total Phenolics	EPA 420.2	WCM-3	EPA 420.2	WCM-8/WCM-29
Total Suspended Solids	EPA 160.2	WCM-1	EPA 160.2	WCM-1
Field Measurements - Groundwater Monitoring				
pH	NA[3]	NA	EPA 150.1	PHT-15670
Temperature	NA	NA	EPA 170.1	PHT-15670
Conductivity	NA	NA	EPA 120.1	SC-15670
Dissolved Oxygen	NA	NA	EPA 360.1	DO-15670
Oxidation-Reduction Potential	NA	NA	SM 2580 B	ORP-15670
Turbidity	NA	NA	EPA 180.1	NTU-15670

[1] VOCs - Volatile Organic Compounds
SVOCs - Semivolatile Organic Compounds
SOP - Standard Operating Procedure

[2] SW-846 - "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods", EPA SW-846, 3rd Edition with promulgated updates, November 1986.
EPA - "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, revised March 1983.
SM - "Standard Methods for the Examination of Water and Wastewater", APHA, 18th Edition, 1992.

[3] NA - Not Applicable

TABLE 8.1

SURROGATE COMPOUNDS PERCENT RECOVERY CONTROL LIMITS
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Analysis	*Matrix*	*Surrogate Compound*	*% Recovery Control Limits* [1]
Volatile Organic Compounds	Water	Bromofluorobenzene	77-133
		Toluene-d_8	76-133
		Dibromofluoromethane	77-130
Semivolatile Organic Compounds	Water	Nitrobenzene-d_5	53-125
		2-Fluorobiphenyl	54-130
		Terphenyl-d_{14}	32-145
		Phenol-d_5	15-56
		2-Fluorophenol	24-83
		2,4,6-Tribromophenol	42-140

[1] Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for data validation purposes.

TABLE 11.1

**ROUTINE PREVENTIVE MAINTENANCE
PROCEDURES AND SCHEDULES
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS**

Instrument	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Gas Chromatograph/Mass Spectrometer (GC/MS)	1. Replace pump oil as needed. 2. Change septa weekly or as often as needed. 3. Change gas line dryers as needed. 4. Replace electron multiplier as often as needed. 5. Replace gas jet splitter as needed. 6. Replace GC injector glass liner weekly or as often as needed. 7. Replace GC column as needed. 8. Check to ensure that gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 9. Check to ensure the pressure on the primary regulator never runs below 100 psi.	1. Syringes 2. Septa 3. Various electronic components 4. Glass jet splitter 5. GC column 6. Glass liners
Gas Chromatograph	1. Change septa weekly or as often as needed. 2. Change gas line dryers as needed. 3. Replace GC injector glass liner weekly or as often as needed. 4. Replace GC column as needed. 5. Clean/replace GC detector as needed. 6. Check to ensure that gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 7. Check to ensure the pressure on the primary regulator never run below 100 psi.	1. Syringes 2. Septa 3. Detectors 4. Glass liners 5. GC column

TABLE 11.1

ROUTINE PREVENTIVE MAINTENANCE
PROCEDURES AND SCHEDULES
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Instrument	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Purge and Trap Sample Concentrator	1. Replace trap as needed. 2. Decontaminate the system after running high concentration samples or as required by blank analysis. 3. Leak check system daily and as often as needed 4. Check to ensure the gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 5. Check to ensure the pressure on the primary regulator never run below 100 psi.	1. Spare traps 2. Spare sparger 3. Various electronic components/ circuits 4. Plumbing supplies - tubing, fittings
Mercury Analyzer	1. Clean tubing and quartz cell weekly or as often as needed. 2. Clean aspirator as necessary. 3. Check to ensure the gas supply is sufficient pressures are set as described in the SOP.	1. Quartz cells 2. Aspirator
Inductively Coupled Plasma Spectrometer (ICP)	1. Clean torch assembly and mixing chamber when discolored or after eight hours of running high dissolved solid samples. 2. Clean nebulizer as needed. 3. Check to ensure the gas supply is sufficient for the day's activity pressures are set as described in the SOP.	1. Spare torch mixing chambers 2. Spare nebulizer

TABLE 11.1

ROUTINE PREVENTIVE MAINTENANCE
PROCEDURES AND SCHEDULES
WCP SITE PILOT PROJECT
WAUKEGAN, ILLINOIS

Instrument	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Ion Chromatograph	1. Change guard column when necessary as indicated by backpressure readings. 2. Clean or replace sample loop as needed. 3. Replace separator column when necessary as indicated by chromatography. 4. Replace suppressor when necessary as indicated by detector response.	1. Guard and separator columns 2. Sample loops 3. Suppressor
Spectrophotometer/ Autoanalyzer	1. Clean cuvettes and tubing after each use or as often as needed. 2. Inspect and clean peristaltic pump and necessary. 3. Check lamp daily and clean as necessary	1. Cuvettes 2. Pump tubing 3. Spare lamps
Specific Ion Meter	1. Check meter function daily using shorting cables. 2. Check electrolyte levels in reference and analytical probes prior to use. 3. Clean probes after each use.	1. Standard solutions 2. Electrolyte filling solution 3. Spare electrodes
Flow-through Conductivity, pH, Temperature, Redox Meter	1. Check battery prior to use and replace as necessary. 2. Rinse flow-through between samples and clean probes with detergent and rinse thoroughly with deionized water .	1. Standard solutions 2. Spare electrodes 3. Spare batteries

TABLE 11.1

ROUTINE PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES WCP SITE PILOT PROJECT WAUKEGAN, ILLINOIS

Instrument	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Dissolved Oxygen Meter	1. Check battery and replace if needed. 2. Check membrane and filling solution prior to each use. 3. Rreplace membrane if torn or wrinkled 4. Polish gold electode if it becomes tarnished.	1. Standard solutions 2. Spare membranes 3. Spare batteries
Electric Water Level Meter	1. Check battery and replace if needed. 2. Check connection between probe and tape periodically. Repair with electrical tape if required. 3. After use, wash the probe and reel in soap and rinse thoroughly with distilled water.	1. Probes, tapes, cable reels, batteries
Turbidity Meter	1. Replace turbidity standards after nine months of use. Store between 10° and 40°C. 2. Do not open the standards in a dusty environment. Do not put unused standard or possible contaminant into opened standard bottle. 3. After each use, rinse turbidity tubes with detergent and distilled water. Replace tubes as required. 4. Replace Nickel-Cadmium batteries as required. Charge batteries after every use.	1. Cuvettes, batteries

APPENDICES

APPENDIX A

PILOT PROJECT WORK PLAN

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NEWFIELDS, INC.

Pilot Project Work Plan

Waukegan Manufactured Gas and Coke Plant, Waukegan, Illinois

May 23, 2000

TABLE OF CONTENTS

1.0 INTRODUCTION

The Waukegan Manufactured Gas and Coke Plant (WCP) site is located in Waukegan, Illinois, on the peninsula separating Waukegan Harbor from Lake Michigan (Figure 1.1). The property and its environs have been part of the industrial/commercial waterfront in Waukegan. The sand dunes and beach area adjacent to the WCP Site are used for public recreation.

The WCP Site is underlain by near-surface fill materials that were placed over a fine-grained sand unit. The sand unit extends from the ground surface to the top of a low-permeability clayey till unit. The shallow groundwater occurs in a 30-foot-thick fine sand unit. Shallow groundwater flows in response to infiltration on the peninsula, discharging to the surrounding surface water.

The vadose zone soil and the deep portion of the shallow aquifer at the WCP Site have been adversely impacted due to past activities. Soil at the WCP Site is contaminated with tar and arsenic. The groundwater is mainly contaminated with arsenic, phenols, ammonia, benzene, cyanide, and thiocyanate. The impacted portion of the shallow aquifer is found in the lowest 5 feet of the sand unit, approximately 25 feet below ground surface. Figure 1.2 shows a plan view of the impacted portion of the shallow aquifer. This figure also shows the location of a beach transect. The vertical extent of arsenic and phenols in the shallow aquifer along the beach transect is illustrated in Figures 1.3 and 1.4.

Upon completion of the Remedial Investigation/Feasibility Study (RI/FS), the Record of Decision (ROD) for the WCP Site was issued in September 1999. The ROD defined six elements of the site groundwater remedy:

1. Short-term groundwater removal and on-site treatment/re-infiltration
2. Groundwater treatment
3. Waiver of the underground injection control prohibition
4. Long-term monitored natural attenuation
5. Long-term monitoring
6. Five-year reviews

The ROD groundwater remedial objectives are divided into two phases, as short-term (Phase 1) and long-term (Phase 2) goals. The short-term goal is a substantial reduction of contaminants at the deep portion of the shallow aquifer in order to remove the chemical inhibitors of natural attenuation, and minimize impacts of contaminated groundwater on Lake Michigan and harbor surface water. Subsequently in Phase 2, the long-term remedial goals are pursued based on Monitored Natural Attenuation. As noted in the ROD: "*Once the inhibitive concentrations of contaminants have been removed and the nitrate source and oxygenation from treatment re-injection is available in the aquifer, degradation should occur.*" In the long-term, attainment of maximum concentration limits (MCLs) is anticipated.

The ROD states that the design of the Phase 1 groundwater remedy will be based in part on pilot testing of a groundwater extraction and re-injection system. This Pilot Project Work Plan focuses on the Phase 1 elements of the groundwater remedy: (1) short-term groundwater removal and onsite treatment/re-infiltration, and (2) groundwater treatment.

This Pilot Project Work Plan is presented in nine sections, titled: (1) Introduction (this section); (2) Pilot Project Objectives and Data Needs; (3) Conceptual Approach; (4) Study Area Characterization; (5) Pilot Extraction and Re-injection Units; (6) Bench-Scale Groundwater Treatment Assessment; (7) Pilot Project Data Analysis Goals; (8) Pilot Project Report Outline; and (9) Pilot Project Schedule. More detailed information concerning the WCP Site characterization and alternative remedies are provided in the RI/FS (Barr, 1995 and 1998).

2.0 PILOT PROJECT OBJECTIVES AND DATA NEEDS

As stated in the ROD, the design and implementation of the selected groundwater remedy (i.e., the mobile, cell-based, low-flow extraction/treatment/re-injection system) will be based on the current RI/FS data, the pre-design investigation, and pilot testing. Consistent with the ROD framework, the objective of this Pilot Project is to determine design parameters and constraints for implementation, operation, and performance measurement of an extraction/re-injection unit of the ROD groundwater remedy.

To attain the objective of the Pilot Project, the following data needs must be met:

A. ***Pilot Study Area Characterization:*** Characterization of the pilot study area is needed to allow extrapolation of the pilot study results over the entire site. For this purpose, the lateral and vertical extents of the groundwater contaminants of concern in the study area will be adequately determined. This data need will be addressed with direct-push probe profiles and data from monitoring wells installed as a component of the extraction pilot testing.

B. ***Hydrogeologic Constraints to Mass Removal/Re-injection:*** The effectiveness of the extraction/re-injection units will be constrained by the hydrogeologic and geochemical characteristics of the impacted portion of the shallow aquifer. During the Pilot Project, these constraints will be determined through direct monitoring of the performance of the pilot units. For this purpose, pilot units will be operated under a variety of scenarios, such as: (1) constant low-flow extraction/re-injection; (2) intermittent (pulse) low-flow extraction; and (3) variable extraction rates. A tracer test will also be conducted during the constant low-flow extraction/re-injection test to better characterize the groundwater flow regime during the operation of the pilot units. Throughout these pilot testing activities, multi-depth groundwater samples will be collected on a regular basis. The resulting data will provide key information on mass removal rates and trends under various extraction scenarios, as well as groundwater flow-regime under low-flow extraction/re-injection process. The analyses of collected data will form the foundation of the design and operation of the field-scale extraction/re-injection units.

C. ***Treatment Constraints/Natural Attenuation Threshold Criteria:*** The ROD groundwater remedy calls for the treatment of the extracted water prior to its re-injection into the deep portion of the shallow aquifer. This treatment is aimed at achieving a two-faceted goal - treating the extracted water for contaminant removal, while yielding geochemical properties to enhance the natural attenuation of the impacted groundwater. As the ROD states, upon completion of the extraction/re-injection phase of the remedy, the long-term groundwater remedial goals will be attained through natural attenuation. Therefore, during the Pilot Project, representative extracted water samples will be the subject of a bench scale treatability study. The bench scale is designed to determine: (1) contaminant removal effectiveness and the limitations of various alternative treatment processes and trains; and (2) the impact of the extraction/re-injection process on reduction of contaminant concentrations at the deep portion of the shallow aquifer. This pilot information, along with previous site-specific experimental and numerical results, may also provide a basis to define the in-situ threshold contaminant concentrations and/or loads within the deep portion of the shallow aquifer beyond which ROD long-term remediation objectives can be attained through natural attenuation.

To address the above Pilot Project objective and data needs, a pilot testing system is proposed. The conceptual aspects of the proposed system are described in the following section.

3.0 CONCEPTUAL APPROACH

The data needs of this Pilot Project require operation of the system under a variety of scenarios. For this purpose, a two-unit system is proposed, as depicted in Figure 3.1. Components of this system are:

A. ***Extraction/Re-injection Unit (E/R Unit):*** This unit is composed of three extraction wells and six re-injection wells. This unit is intended to simulate the simultaneous operation of low-flow extraction and re-injection wells. In such units, the outer re-injection wells are intended to supply flushing water that may enhance the removal efficiency of the inner extraction wells. The E/R Unit will be operated at a constant extraction rate for the duration of the pilot testing period. During the Pilot Project, tap water will be used for re-injection. Periodically during the operation of the E/R Unit, the tap water will be sampled for pH, chlorine, and dissolved oxygen to verify the quality of the injected water and assess any impacts on the re-injection process.

B. ***Extraction Unit (E Unit):*** This unit is composed of a single extraction well, which will be operated under both steady state and pulse conditions with up to three different extraction rates. The data from this unit, as well as the E/R Unit, will provide a comparative basis to determine effective extraction/re-injection operation patterns, rates, and scheduling. Specifically, removal efficiency of extraction wells will be evaluated under constant versus intermittent (pulse) operation, as well as different extraction rates.

C. ***Equalization Tanks:*** As depicted in Figure 3.1, the extracted water from both units will be stored in three 20,000-gallon Equalization Tanks. These tanks will be used to provide short-term storage for the extracted water during the Pilot Project, and may be used for quality/flow equalization during the operation of the full-scale treatment system. If used during the operation of the full-scale system, the tanks would enhance the effectiveness of the system by equalizing wide concentration variations during operation of an extraction/re-injection cell. The Equalization Tanks can also serve as separators in the event of observing non-aqueous phase liquids in the extracted water. The treatability study will be conducted based on water samples from the Equalization Tanks. The remaining water stored in these tanks is intended to be either used as influent for the initial start-up operation of the future onsite treatment system, or disposed of offsite.

More detailed information concerning the elements of the pilot study is provided in the following sections.

4.0 STUDY AREA CHARACTERIZATION

The pilot study area is shown in Figure 1.2. Further details about the configuration of Units within the study area are provided in Section 5.1. Characterization of the study area will be conducted the following testing:

1. At least, two direct-push or GeoProbe vertical geophysical profiles will be collected to estimate the vertical extent of the impacted portion of the shallow aquifer. Geophysical profiles will be collected close to the center of each Unit using Cone Penetrometry Gas Chromatography. This technology will be used to create a profile of both the bulk organic contaminant concentration and the bulk density of the soil with depth.

2. Groundwater samples from the deep portion of shallow aquifer will be collected from the *extraction and re-injection wells prior to initiation of the testing . Proposed* sample analyses are described in Section 5.3.
3. Multi-depth groundwater samples will be collected at two installed monitoring well nests associated with the E/R Unit and at one installed monitoring well nest associated with the E Unit. These clustered wells will be installed using the micro-well or direct-push technology. Proposed sample analyses are described in Section 5.3.

5.0 PILOT EXTRACTION AND RE-INJECTION UNITS

5.1 PHYSICAL DESCRIPTION

Consistent with the findings of the FS (Barr, 1998) and the ROD-selected short-term remedy, two groundwater extraction units will be installed during the Pilot Project, denoted as the E/R and E Units[1]. The E/R Unit will consist of nine wells laid out in three parallel rows with one extraction well and two re-injection wells in each row. A plan view of the E/R Unit is shown in Figure 5.1. A transect across the E/R row at the center of the E/R Unit is shown in Figure 5.2. Each extraction well will be screened in the bottom 5 feet of the shallow aquifer. The re-injection wells will be screened in the bottom 5 to 10 feet of the shallow aquifer, depending on the thickness of the impacted portion of the aquifer. Water extracted from the inner three extraction wells will be stored in onsite Equalization Tanks. Tap water will be re-injected in the outer six wells. During the pilot testing, the E/R Unit wells will be operated at constant extraction and re-injection rates of approximately 0.3 gallons per minute (gpm) and 0.15 gpm per well, respectively. The wells will be controlled individually to balance extraction and injection flows among the wells.

The second test unit (the E Unit) will consist of a single extraction well screened in the bottom 5 feet of the shallow aquifer. Similar to the E/R Unit, the extracted groundwater

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[1] Investigative-derived soil waste (e.g., drill cuttings) will be placed in drums. These drums will be disposed along with the RI/FS-related waste drums that are currently located onsite.

from this unit will be stored in onsite Equalization Tanks. The E Unit will be operated intermittently at variable extraction rates, as discussed in the following subsection.

5.2 OPERATIONAL DESCRIPTION

Consistent with the ROD-selected short-term remedy, based on a low-flow, cell-based extraction/re-injection system, the E/R Unit will be pumped at a constant low-flow rate of approximately 0.9 gpm (i.e., 0.3 gpm from each extraction well) for approximately 4 weeks. Simultaneous with groundwater extraction, 0.9 gpm of tap water will be injected into the re-injection wells (i.e., 0.15 gpm into each re-injection well).

At the initiation of the operation of the E/R Unit, a bromide tracer test will be conducted. For this purpose, bromide will be added to the re-injected tap water upon commencement of operation of the central re-injection well closest to the monitoring well nest. Subsequently, groundwater samples will be analyzed from monitoring, extraction and re-injection wells to determine the path and rate of groundwater flow between the re-injection and extraction wells.

The E Unit will undergo an intermittent extraction schedule with the pump on for 7 days and then off for 7 days. Four cycles are contemplated for the pilot testing. The extraction rate from the E Unit will be reduced with each successive pumping cycle, starting at 0.8 gpm and ending at 0.2 gpm. The extraction schedule and rates for both units are presented in Table 5.1.

5.3 SYSTEM MONITORING

Groundwater quality will be monitored within the E/R Unit using two nests of five monitoring wells. A plan view of the monitoring well placement is shown in Figure 5.1. The multi-depth monitoring well nest 1 is located in a point that is expected to be highly affected by the flow generated by the operation of the extraction and re-injection wells. Nest 2, on the other hand, is situated between two extraction wells, which could create a nearly stagnant condition in the vicinity of this latter nest of monitoring wells. Therefore, the monitoring data from the two nests would provide information on the entire range of removal effects of the E/R Unit.

Each monitoring well will be screened over an interval not to exceed 12 inches, as indicted in Figure 5.2. Groundwater quality in the vicinity of the E Unit will be monitored using a single nest of five monitoring wells, as shown on Figure 5.3. The nest of monitoring wells will be set approximately 5 ft from the E Unit well. These monitoring well nests will be installed using the micro-well or direct-push technology. All water samples will be collected with minimal purging[2]. The sampling technique to be used will entail inserting a small diameter tube down the monitoring well, purging only the volume of the tube, and then collecting the sample. This technique will minimize the influence of the sample volume on in-situ contaminant concentrations. Collected groundwater samples will be routinely analyzed for field parameters, including pH, temperature, chloride, and dissolved oxygen. Groundwater levels may also be measured as part of the Pilot Project monitoring efforts. The scopes of chemical analyses on each sample are presented in Table 5.2.

During the operation of the two Units the following sampling activities will be conducted:

1. ***Monitoring Wells***: Sampling and analysis of the monitoring wells within the E/R and E Units will be conducted according to the schedule specified in Table 5.2. In the E Unit, two of the scheduled samples each week will be drawn on the same day that the pump operational mode is changed (i.e., pumping started or stopped).
2. ***Tap Water Testing:*** Tap water, which will be re-injected during the operation of the E/R Unit, will be periodically sampled and analyzed for pH, chlorine, and dissolved oxygen.
3. ***Tracer Test:*** Bromide tracer sampling of the monitoring wells within the E/R Unit will be conducted as specified in Table 5.2. Monitoring wells along with extraction and re-injection wells of the E/R Unit will be sampled daily for bromide for a period of 7 days. Bromide sampling will then shift to three times per week for the remainder of the E/R Unit test.
4. ***Extracted Water:*** Sampling of the extracted water from each extraction well of E/R and E Units will be conducted three times per week. The sampled water will be analyzed for the parameters identified in Table 5.2. In the E Unit, at least one sample each week will be drawn on the same day that that the pump operational mode is

[2] Investigative-derived water waste (e.g., purged waters) will be placed in the Equalization Tanks.

changed (i.e., pumping started or stopped). One sample will also be drawn at the midpoint of an operational mode.

5. ***Real Time Monitoring:*** Specific conductance of the outflow of the central extraction well of the E/R and E Units will be continuously monitored during the Pilot Project to monitor short-term variations in the quality of the extracted water.
6. ***Pilot Project Post-Extraction Monitoring:*** The extraction wells within the E/R and E Units will be sampled one week and one month after completion of testing to assess the rate of recovery of contaminants at the Pilot Project Units. The sampled water will be analyzed for parameters identified in Table 5.2.

Due to the frequency of the sampling, the advantages of minimizing sample volume, and the expected continuity in concentrations, duplicate samples are not needed in the above monitoring efforts. Upon availability of the above data, subsequent post-Pilot-Project monitoring may be planned and conducted to further assess the long-term effects of the low-flow extraction/re-injection system. All monitoring, extraction and re-injection wells that are deemed unnecessary for further sampling or full-scale implementation of cell-based remedy will be abandoned.

6.0 BENCH-SCALE GROUNDWATER TREATMENT ASSESSMENT

6.1 PROCESS WATER PRE-TREATMENT

Extracted water from the E Unit will be stored in 20,000-gallon tanks (i.e., the Equalization Tanks) onsite. Once a tank is filled, 75 gallons of the equalized groundwater will be drawn from the center of the tank. This water will be treated using the ANDCO[3] electro-chemical precipitation technology for arsenic removal using electro-chemical precipitation. The arsenic removal operating parameters will be based on the results of arsenic removal testing during the RI and the arsenic concentration in the process water. The treated water

[3] ANDCO Environmental Processes, 595 Commerce Drive, Buffalo, NY 14228. Telephone: (716) 691-2100

will be sampled to verify greater than 90% of the arsenic[4] is removed prior to shipping the treated water to a laboratory for biological treatment.

6.2 BENCH-SCALE BIOLOGICAL TREATMENT TESTING

The bench-scale biological treatment test will consist of at least two separate treatment trains, as described below:

1. The first treatment train will consist of two aerobic sequencing batch reactors (SBR) in series. The first SBR (SBR-1) will be operated to achieve biological degradation of organic compounds. The second SBR (SBR-2) will be operated to convert ammonia to nitrate (nitrification).
2. The second treatment train will consist of a single aerobic SBR (SBR-3) operated to achieve both organic removal and nitrification using the same sludge.

Additional treatment trains may also be considered.

Seed sludge for each SBR will be obtained from a full-scale activated sludge treatment system that treats coke plant wastewater and achieves both biological organic removal and nitrification (e.g., US Steel-Gary Works). The influent to SBR-1 and SBR-3 will be the groundwater pre-treated for arsenic removal. The influent to SBR-2 will be the effluent from SBR-1.

During start-up, all of the SBRs will be operated on 6-hour cycles. The duration of each period within each cycle is presented in Table 6.1. The initial operating parameters for the SBRs are provided in Table 6.2. The values of these parameters are based on a pilot scale test of the biological treatment of coke plant wastewater (ref. Rupnow, Shelby, Singh, "Development of a New Wastewater Treatment System for a Major Coke Plant", Proc. Water Environment Federation 70th Annual Conference and Exposition, Chicago, Illinois, vol 3, part 2, pp. 265-276, 1997) . Each SBR will be operated continuously for four cycles each day for a minimum of one solids retention time (SRT). Table 6.3 presents the daily analysis

[4] Final arsenic removal rate during the full-scale onsite treatment of extracted water will be determined based on site-specific data. For example, during the future natural attenuation study, as envisioned by the ROD, the effects of arsenic concentration on in-situ biodegradation will be addressed, which could lead to a different arsenic removal rate.

to be accomplished during this acclimation phase. All daily analysis will be performed during the same cycle. At the end of this phase of testing, performance verification samples will be drawn for analyses, as presented in Table 6.4. These samples will be collected during one cycle each day for three consecutive days.

Once a SBR has operated for at least one SRT, the variation of parameters during a single cycle will be determined. During a single cycle of a SBR, the fill period (FILL) will be reduced to less than 5 minutes with no aeration. Once FILL is complete, the SBR will be mixed without aeration, and an initial sample will be collected. After sampling, the aerated react period (REACT) will start and the cycle will proceed using the operating strategy outlined in Table 6.1. The list of samples to be drawn and the analysis parameters for these batch time-based studies are presented in Table 6.5.

7.0 PILOT PROJECT DATA ANALYSIS GOALS

This section describes the goals of the analysis of the Pilot Project data. Graphical and statistical techniques will be employed to assess the variations in groundwater quality parameters during different phases of the Pilot Project. These analyses will be the basis for determining design parameters and constraints for implementation, operation, and performance measurement of extraction/re-injection cell units. These extraction/re-injection units constitute the short-term component of the ROD groundwater remedy.

7.1 HYDROGEOLOGIC DATA ANALYSIS GOALS

The chemical data collected prior to and during the operation of the E/R and E Units will be analyzed to address the following design issues, as listed below.

A. ***Effective Full-Scale Groundwater Characterization:*** The geophysical profiles will be produced during the characterization of the Pilot Project study area. The comparison of these profiles with monitoring well nest data will determine the applicability of the use of the geophysical methods for the full-scale, vertical characterization of the groundwater zone, which has been targeted for cell-based extraction and re-injection remedy (Figure 1.2). The combination of such field tests along with focused groundwater sample analyses can provide an effective alternative for groundwater quality characterization of the targeted zone.

B. ***Removal Rate/Concentration Decay in E/R Unit:*** Time series plots of collected groundwater quality data at various depths and locations, as well as extracted water measured concentrations, will be analyzed to estimate the contaminant mass removal, concentration decay rates, and removal limitations under full-scale operation. This analysis will be used to establish groundwater extraction termination criteria.

C. ***Impacts of Re-injection:*** Through comparison of the time series groundwater quality data collected at the E/R and E Units, the impact of re-injected water will be assessed. The re-injected water may enhance the restoration of the groundwater. Specifically:

- The flushing/sweeping effects of the re-injected water could increase the effectiveness of the inner extraction wells in the removal of contaminants.
- The re-injection of the treated water could reduce concentrations of attenuation inhibitors, and thus, enhance the rate of in-situ natural attenuation of groundwater contaminants.
- The chemical characteristics of the re-injected water, such as higher oxygen and nitrogen contents, could further accelerate the natural attenuation of groundwater contaminants.
- The re-injection of water could also cause local dispersion of groundwater contaminants toward the upper portion of the shallow aquifer. As supported by site-specific data (e.g., Figures 1.3 and 1.4), such dispersions may yield a more rapid degradation of contaminants in the upper portion of the shallow aquifer.

Over time, however, the dilution caused by re-injection of treated water can gradually reduce the mass removal efficiency of an extraction unit. In other words, re-injection may gradually reduce the mass of contaminants per unit volume of extracted water.

Comparison of the E/R and E Units removal performance will provide information on appropriate re-injection schemes. The intent is to increase the positive effects of re-injection, while minimizing effects of gradual removal efficiency decreases. The analysis will consist of the following:

- Comparison of the mass removal rates over time between the E/R and E Units will determine if removal efficiencies increase or decrease significantly as re-injected water reaches the extraction wells. The results of the bromide tracer testing will be utilized to estimate re-injection water travel times.
- Comparison of the water quality variation and bromide tracer testing results within different zones of the shallow aquifer will be utilized to determine the vertical and horizontal transport of contaminants of concern.

D. ***Impact of Extraction Rate:*** The comparison of the E/R and E Units contaminant removal performance will provide information for determining an appropriate extraction rate within the low-flow range of approximately 0.8 to 0.2 gpm per well. The analysis will consist of comparing mass removal to groundwater removal volumes and estimating the time periods required to reach various target in-situ contaminant concentrations.

E. ***Impact of Cyclic versus Continuous Extraction:*** The data on performance of the continuously operated E/R Unit versus the intermittently-operated E Unit will provide information on assessing the impact of cyclic and continuous extraction on the removal efficiency of an extraction/re-injection system. As with the analysis of extraction rates, the focus of this analysis will be on mass removal relative to groundwater removal volumes and estimation of time periods required to reach various target in-situ contaminant concentrations.

F. ***Effects of Sorption/Desorption:*** Finally, the data during the intermittent operation of the E Unit and the post-extraction sampling will provide information for estimating the effects of sorption, desorption, and transport of various groundwater contaminants on the overall removal efficiency of an extraction/re-injection system.

This analysis will assist in establishing criteria for cycling of groundwater extraction as well as criteria for termination of extraction within a given cell.

7.2 TREATMENT ASSESSMENT DATA ANALYSIS GOALS

The bench-scale groundwater treatment testing data will be used to accomplish three goals, as described below.

A. ***Contaminant-specific Removal Efficiency:*** The first goal is to determine the design removal efficiency for arsenic, phenol, cyanide, and thiocyanate and the nitrification efficiency. The three sets of data collected at the end of the acclimation phase of testing will be used to perform mass balances on the SBRs for each of these compounds. Computed mass balances will be used to calculate the removal efficiencies for each of the compounds of interest.

B. ***Selected Approach for Phenol Degradation and Nitrification:*** The second goal is to select the approach for achieving phenol degradation and nitrification. Both the removal efficiencies and the kinetic data for the SBR-1 and SBR-2 treatment train and SBR-3 will be compared in order to evaluate the merits of each approach for removing the contaminants of concern from the contaminated groundwater.

C. ***Design Parameters:*** The third goal is to determine the kinetic parameters to be used in design of the full-scale groundwater treatment system. Data from the batch test will be utilized to calculate the stoichiometric and reaction rate coefficients for the degradation of each contaminant of concern. These coefficients will then be used to develop kinetic models to be used in the full-scale design.

7.3 DATA ANALYSIS DECISIONS

The results of the Pilot Project data analyses will be used to make design decisions, including:

A. ***Spatial Configuration of Mobile Cells:*** This would include the vertical and horizontal configuration of the extraction and re-injection wells within each full-scale E/R cell.

B. ***Effective E/R Rates:*** An effective extraction and re-injection rate and schedule that enhances the removal efficiency of the E/R cell, while minimizing the adverse effects of the re-injection process, will be determined.

C. ***Simultaneous and Sequential E/R Cell Grouping:*** Based on the Pilot Project data analysis, effective operation strategies for mass removal, treatment, and re-injection will be determined. The operating programs may include simultaneous E/R cell operations, as well as sequential operation of groups of cell in order to maintain the consistency of the treatment unit influent chemical properties. Furthermore, to balance the positive and adverse effects of re-injection on the overall mass removal efficiency, various extraction and re-injection patterns will be evaluated. These patterns may include simultaneous (i.e., same-cell) extraction and re-injection, or offset extraction and re-injection schedules.

D. ***Cell Performance Standards Verification:*** Based on the collected data, appropriate performance standards and goals for cell operation will be developed. These targets include performance standards based on concentration or mass removal of contaminants at the base of the shallow aquifer, extraction volumes, and/or attainment of natural attenuation threshold levels[5], if applicable, subject to site-specific hydrogeologic and treatment constraints. The monitoring plan of each E/R cell, including termination rules and procedures, will also be developed as part of the verification process.

E. ***Treatment System Components:*** The treatability data results will be used to determine various components of the future onsite treatment unit. The selected treatment trains will focus on: (1) achieving treatment mass removal, (2) creating conditions leading to the attainment of natural attenuation threshold levels at the base of the shallow aquifer, if applicable, and (3) benefiting from potential benefits of added nitrate and oxygen.

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[5] Concentrations of natural attenuation inhibitors beyond which ROD long-term remedial objectives can be achieved through natural attenuation processes.

F. ***Treatment Performance Standards Verification:*** Treatment performance standards and goals will be developed based on effluent concentrations, mass removal, and/or attainment of natural attenuation threshold levels at the base of the shallow aquifer, if applicable, subject to hydrogeological and treatment constraints.

8.0 PILOT PROJECT REPORT OUTLINE

The results of the Pilot Project will be documented in a report, which will be submitted to U.S. Environmental Protection Agency (EPA) and Illinois Environmental Protection Agency (IEPA) for review and comment. This report will address the following topics:

1. Description of Pilot Project components and analytical results;
2. E/R cell configuration, including: well configuration, depth, and E/R rates and schedule;
3. Performance standards for E/R cell operation, based on in-situ concentrations, mass removals, extraction volumes, or attainment of natural attenuation threshold levels, subject to hydrogeologic and treatment constraints;
4. Performance standards for treatment unit operation, based on effluent concentration, mass removal, or in-situ attainment of natural attenuation threshold levels, subject to hydrogeologic and treatment constraints; and
5. Performance standard measurement for both cells and treatment unit operation, including monitoring plans, as well as termination rules and procedures.

9.0 PILOT PROJECT SCHEDULE

Upon submittal and approval of this Pilot Project Work Plan the following phases must be implemented:

1. Preparation of Plans, Field Construction Drawings, Treatability Test Protocol, Quality Assurance Project Plan (QAPP), and Sampling Analysis Plan (SAP)
2. Contractor Procurement and Mobilization

3. Installation of Pilot Units and Equalization Tanks
4. Pilot Unit Operations
5. Follow-up Laboratory Treatability Testing of Equalized Extracted Water
6. Pilot Testing and Treatability Study Data Compilation
7. Preparation of Pilot Report

The Pilot Project anticipated schedule table is shown in Figure 9.1.

10.0 REFERENCES

Barr Engineering Company, 1995. Remedial Investigation Report, *Waukegan Manufactured Gas and Coke Plant Site, Waukegan, Illinois*.

Barr, 1998. Feasibility Study, *Waukegan Manufactured Gas and Coke Plant Site, Waukegan, Illinois.*

Table 6.1 Initial SBR Operating Strategy

Period	Duration (hours)
Aerated FILL	2
Aerated REACT	2.5
SETTLE	1
DRAW	0.5

Table 5.2 Groundwater Sampling and Analysis Plan

Cell	Test	Monitoring Well Nest	sampling frequency	Number of events	locations sampled	vertical points/ location	Samples per event	Total Samples	Analysis														
									Total Phenol	As	Ammonia	Bromide	VOC	GC/MS (Base/Neutral)	GC/MS (Acid)	ORP	Nitrate	COD	Cyanide	Thiocyanate	Alkalinity	TSS
E/R Cell	Contaminant mass removal determination	1	daily	7	1	5	5	35	35	35	35					35						
		1	3Xweek	9	1	5	5	45	45	45	45					45						
		2	3Xweek	12	1	5	5	60	60	60	60					60						
		1 & 2	weekly	4	2	5	10	40					40	40	40	40			40	40		
		1 & 2	start/stop	2	2	5	10	20	20	20	20		20	20	20	20			20	20		
	Subtotal			34	7	25	35	200	160	160	160	0	60	60	60	200	0	0	60	60	0	0
	Tracer Test		daily	14	1	5	5	70				70										
			3xweek	6	1	5	5	30				30										
	Subtotal			20	2	10	10	100				100										
	Extracted water		*3xweek*	*12*	*3*	*1*	*3*	*36*	*36*	*36*	*36*	*36*	*36*	*36*	*36*	*0*	*36*	*36*	*36*	*36*	*36*	*36*
	Subtotal			12	3	1	3	36	36	36	36	36	36	36	36	0	36	36	36	36	36	36
E Cell	Contaminant mass removal determination	3	3Xweek	12	1	5	5	60	60	60	60					60						
		3	weekly	4	1	5	5	20					40	40	40	20			40	40		
		3	start/stop	2	1	5	5	10	10	10	10		10	10	10	10			10	10		
	Subtotal			18	3	15	15	90	70	70	70	0	50	50	50	90	0	0	50	50	0	0
	Extracted water		3xweek	12	1	1	1	12	36	36	36	36	36	36	36	0	36	36	36	36	36	36
	Subtotal			12	1	1	1	12	36	36	36	36	36	36	36	0	36	36	36	36	36	36
Post-Extraction	Contaminant recovery determination (E Wells)		Twice, one-week after, and one-month after	2	4	1	4	8	8	8	8		8	8	8				8	8		
Total								446	310	310	310	172	190	190	190	290	72	72	190	190	72	72

Table 5.1 E/R and E Cell Operation Plan

Test Cell	Pumping Rate (gpm)							
	Week in Test							
	1	2	3	4	5	6	7	8
E/R	0.9	0.9	0.9	0.9				
E	0.8		0.6		0.4		0.2	

Table 6.2 Initial SBR Operating Parameters

SBR	Hydraulic Retention Time (hours)	Solids Retention Time (days)	Average Dissolved Oxygen (mg/L)	Mixed Liquor Suspended Solids (mg/L)
1	24	6 - 10	> 2	2000 - 3000
2	24	15 - 20	> 2	3000 - 5000
3	24	15 - 20	> 2	3000 - 5000

Table 6.3 Sampling and Analyses for Acclimation Monitoring

Sample Location	Time of Sample	Analyses				
		COD	Total Phenol	NH_3-N	MLVSS	pH
Feed Container	FILL	X	X	X		X
SBR Bulk Liquid	REACT				X	X
	DRAW	X	X	X		

COD - chemical oxygen demand

MLVSS - mixed liquor volatile suspended solids

Table 6.4 Samples and Analyses for Performance Verification

Sample Location	Time of Sample	Analyses										
		COD	Total Phenol	Arsenic	NH_3-N	NO_3-N	VOC	GC/MS (Base/Neutral)	GC/MS (Acid)	Cyanide	Thiocyanate	pH
Feed Container	FILL	X	X	X	X	X	X	X	X	X	X	X
SBR Bulk Liquid	Before FILL	X	X	X	X	X	X	X	X	X	X	X
	DRAW	X	X	X	X	X	X	X	X	X	X	X

COD - chemical oxygen demand

Table 6.5 Samples and Analyses for Batch Time Study

Sample Location	Time of Sample	COD	Total Phenol	Arsenic	NH_3-N	NO_3-N	VOC	GC/MS (Base/Neutral)	GC/MS (Acid)	Cyanide	Thiocyanate	Dissolved Oxygen	pH
		Analyses											
SBR Bulk Liquid	After FILL	X	X	X	X	X	X	X	X	X	X		X
	Every 30 min.	X	X	X	X	X	X	X	X	X	X	X	X

COD - chemical oxygen demand

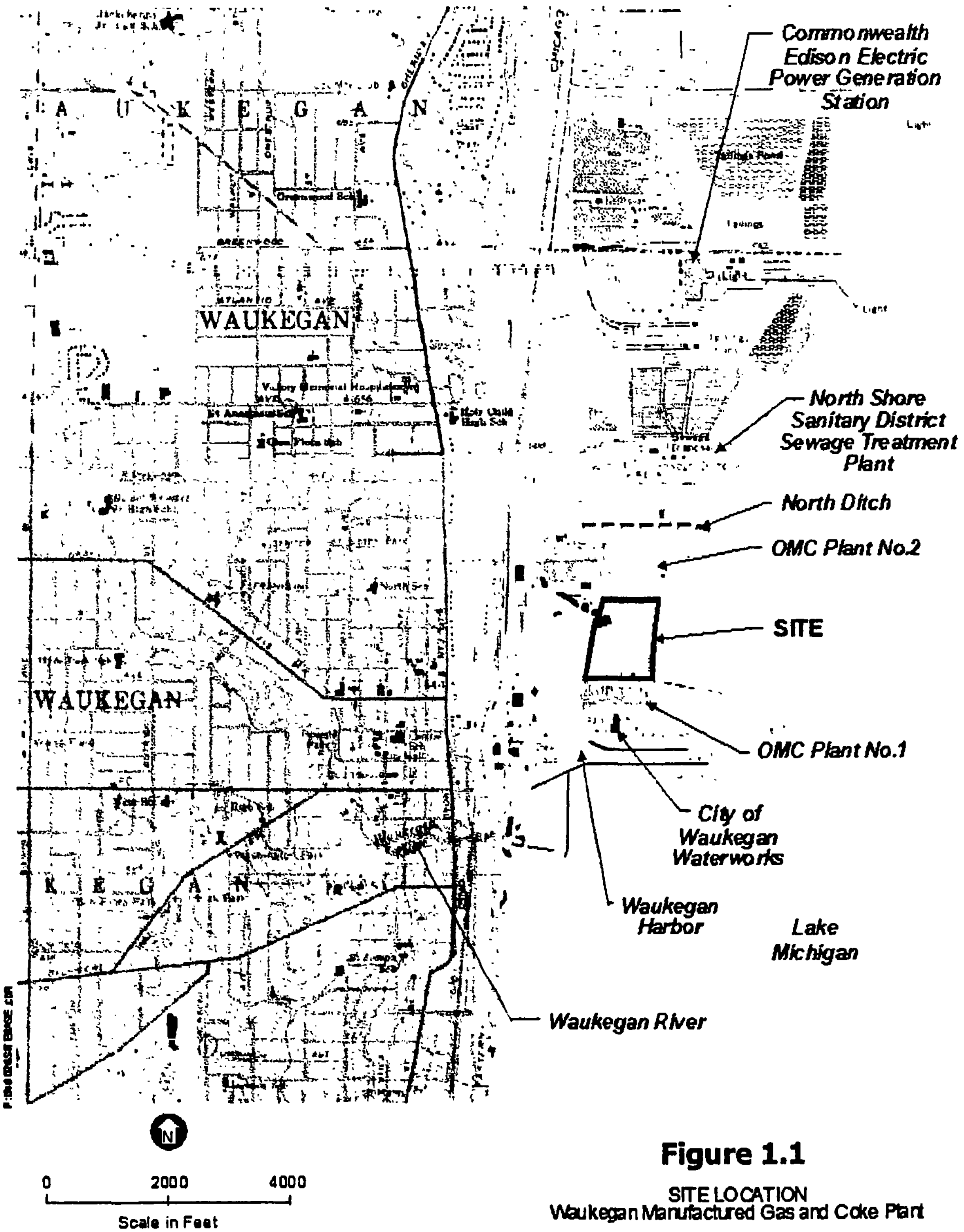
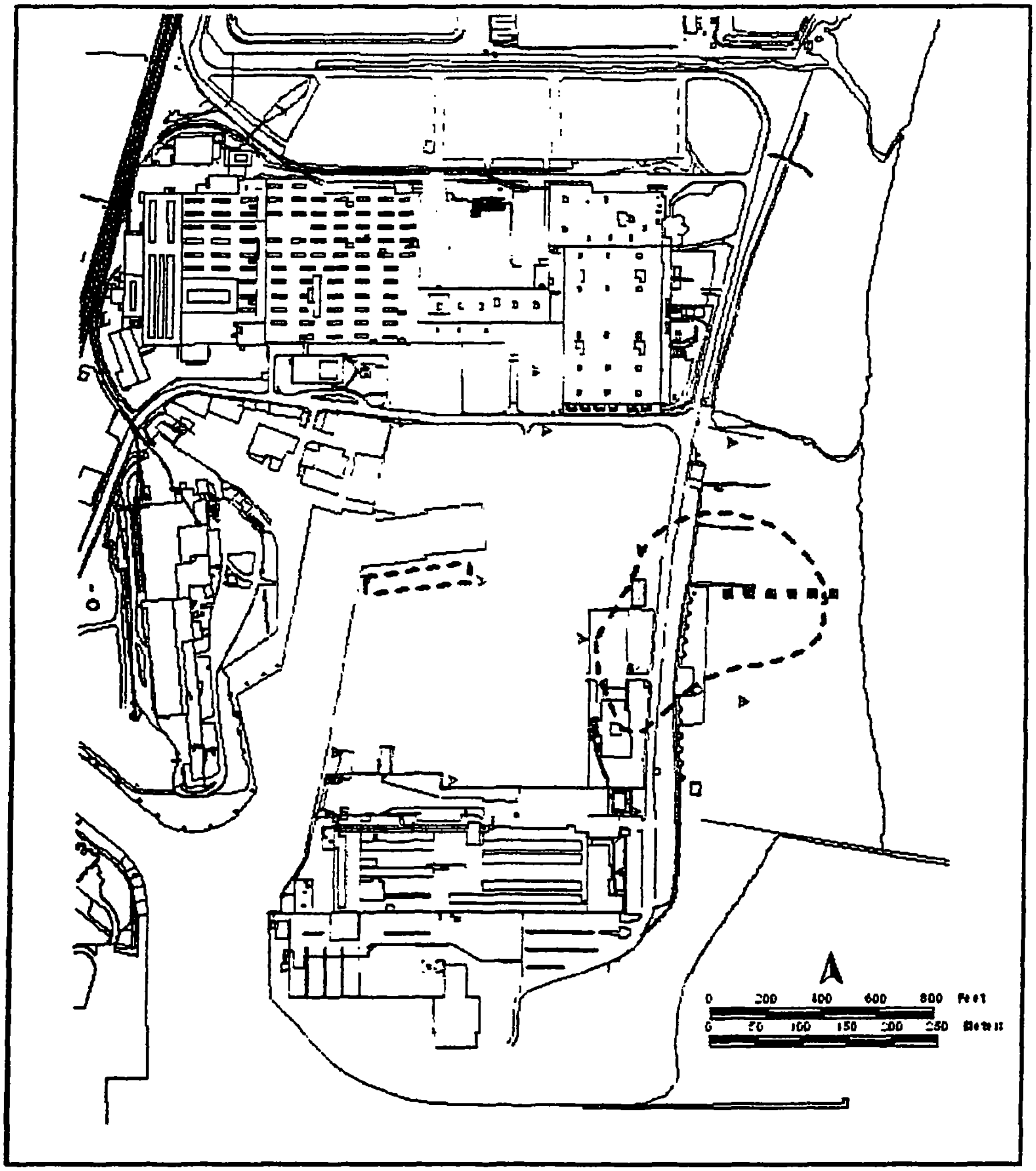


Figure 1.1

SITE LOCATION
Waukegan Manufactured Gas and Coke Plant



Pilot Project Location

Groundwater Remediation Zone Targeted for Treatment Cell Implementation

1997 Beach Transect

Figure 1.2

GROUNDWATER TREATMENT ZONE AND PILOT PROJECT LOCATION

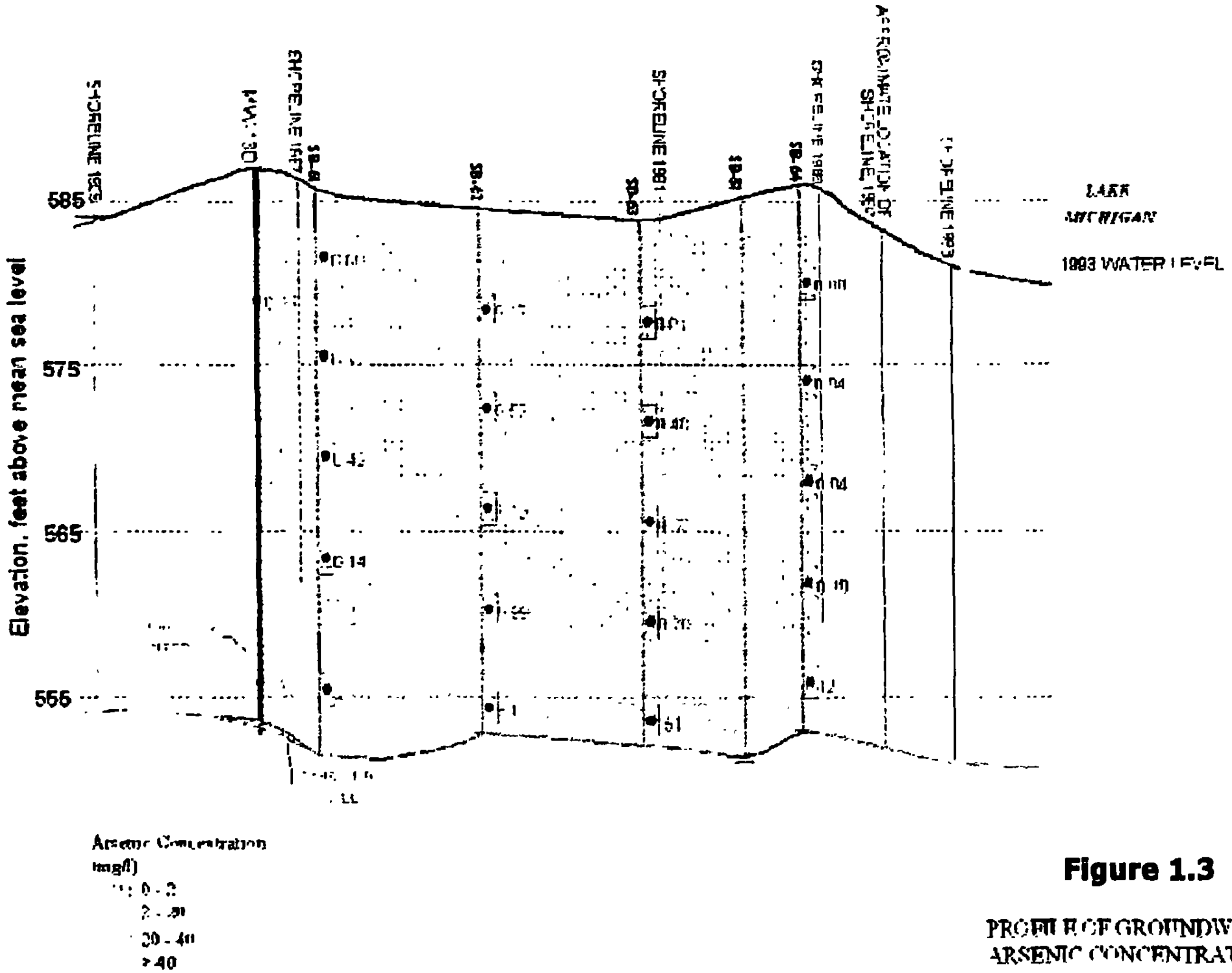
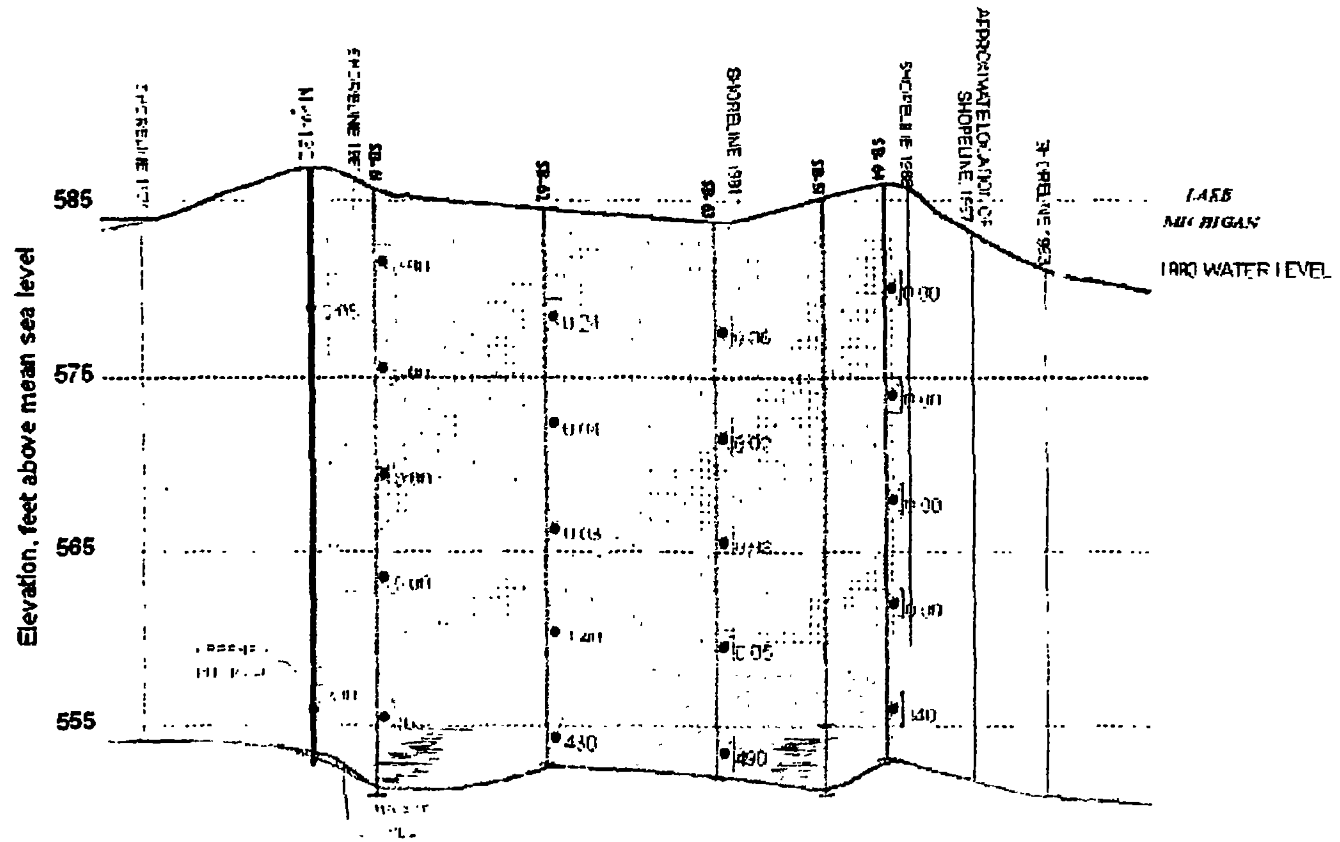


Figure 1.3

PROFILE OF GROUNDWATER ARSENIC CONCENTRATIONS



Phenol Concentration (mg/l)
0 - 1
1 - 10
10 - 100
100 - 400
> 400

Figure 1.4

PROFILE OF GROUNDWATER PHENOL CONCENTRATIONS

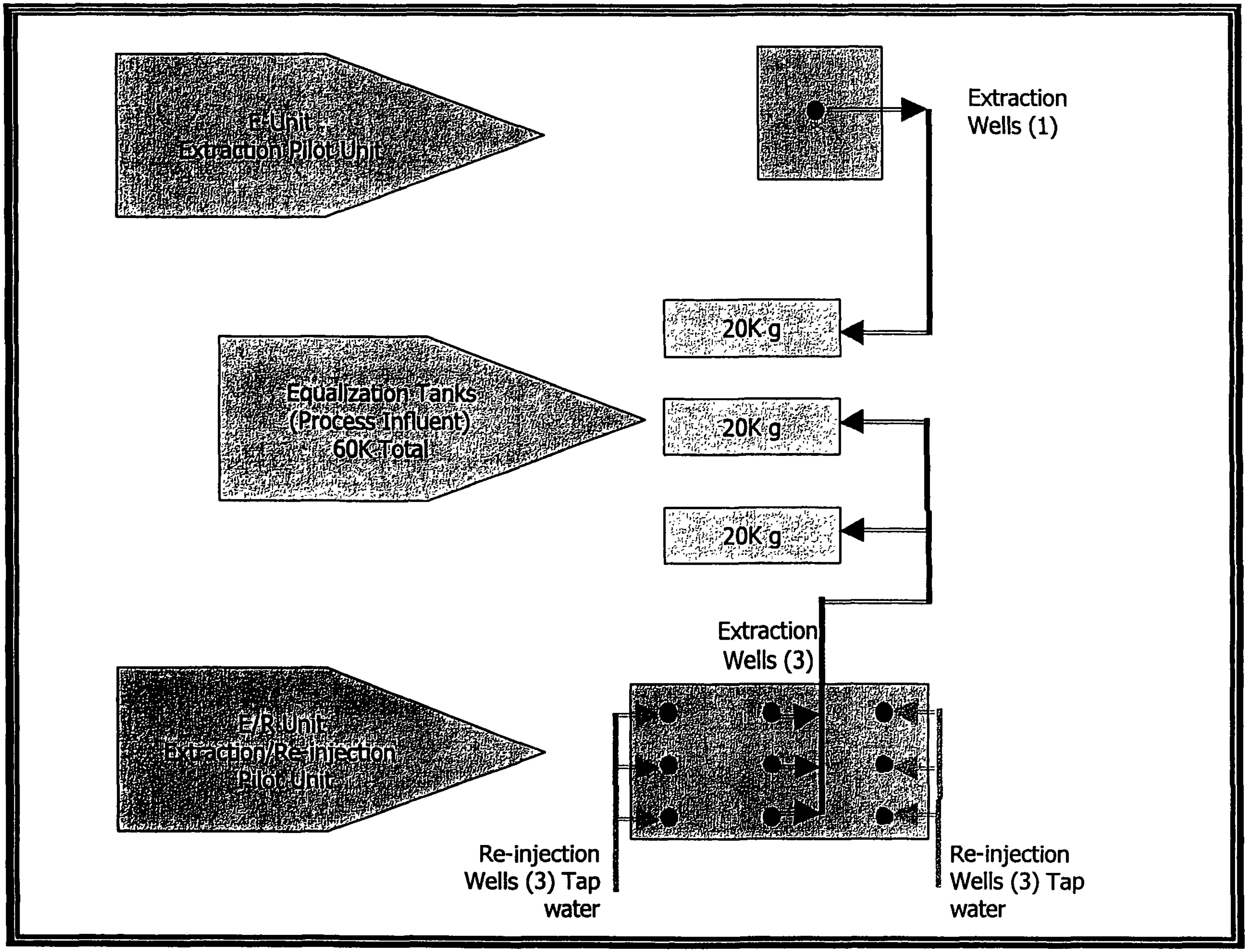
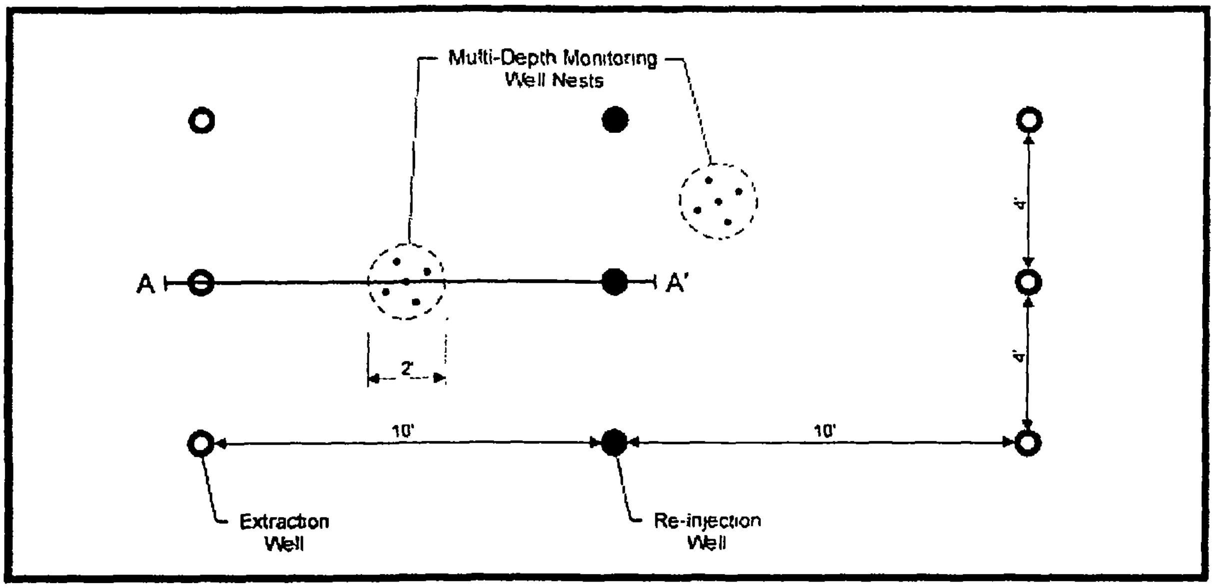
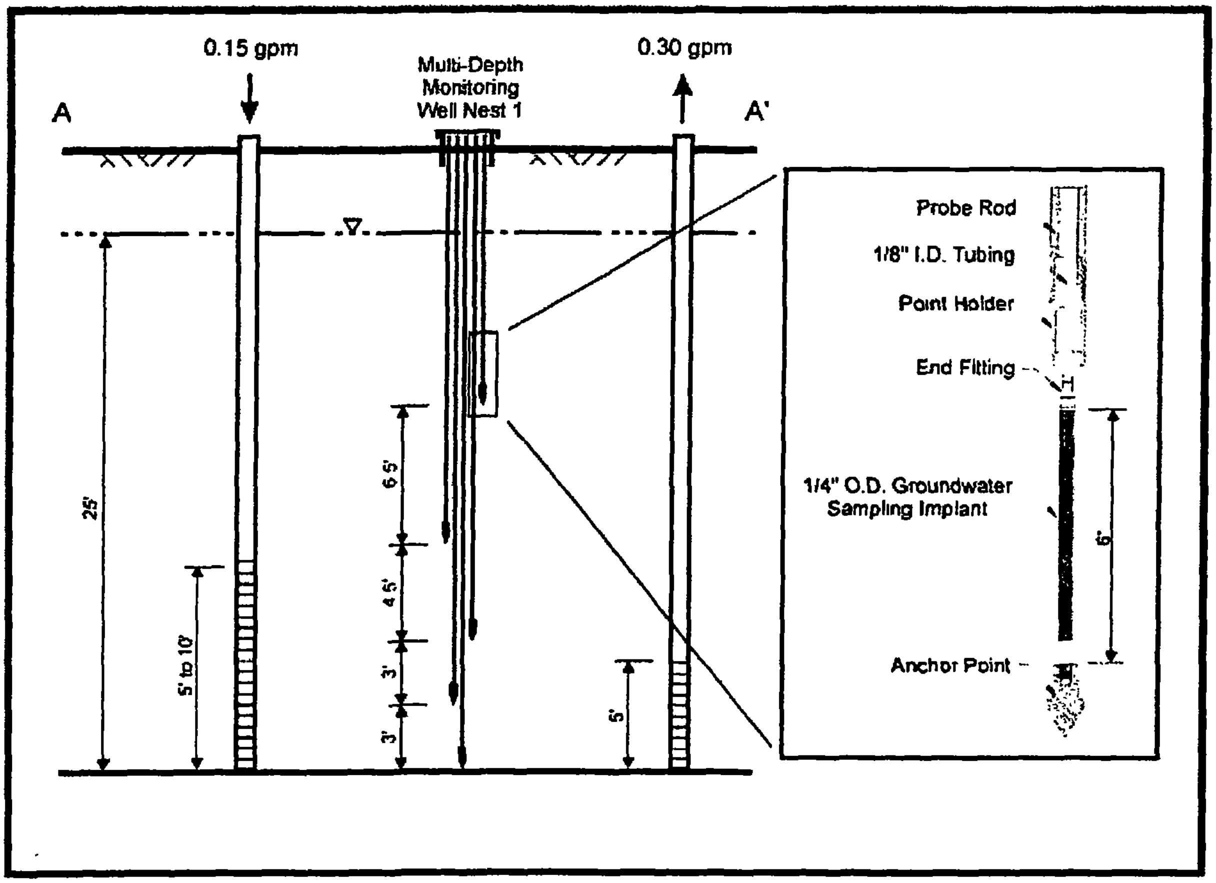


Figure 3.1

Pilot Project Conceptual Configuration



E/R Unit Plan View



E/R Unit Cross-Sectional View

Figure 5.1 E/R Unit

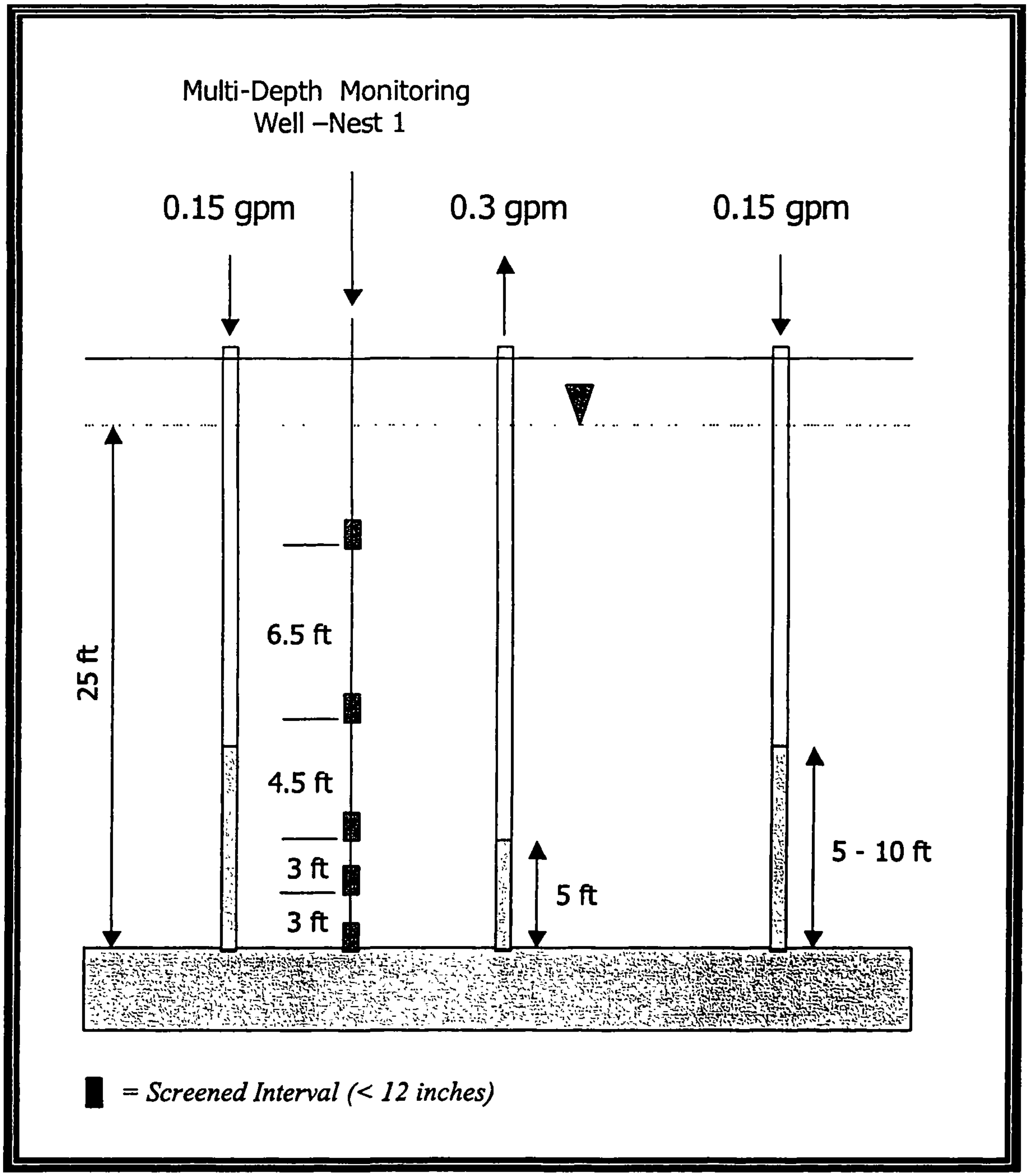


Figure 5.2 E/R Unit Cross Section

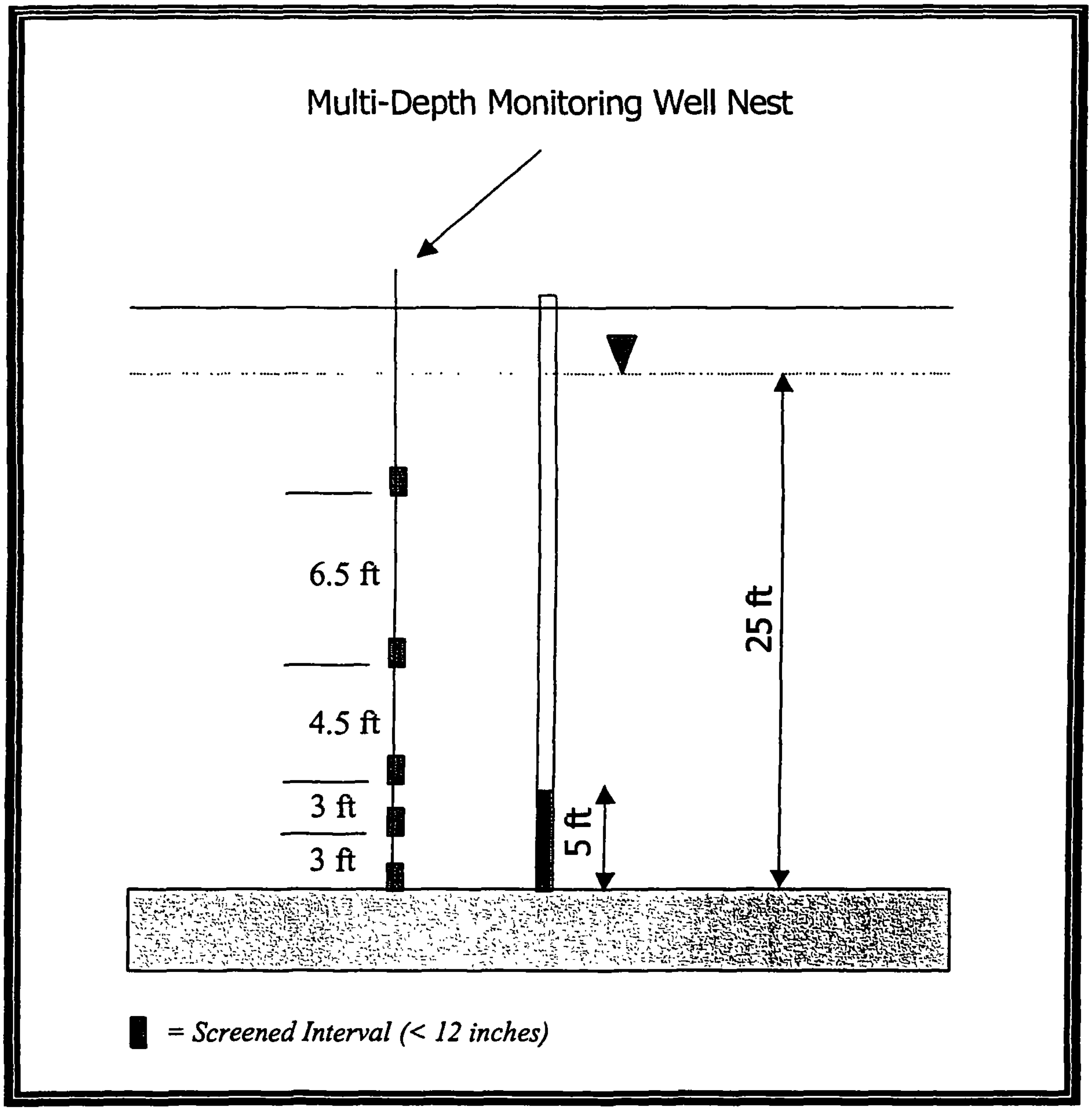
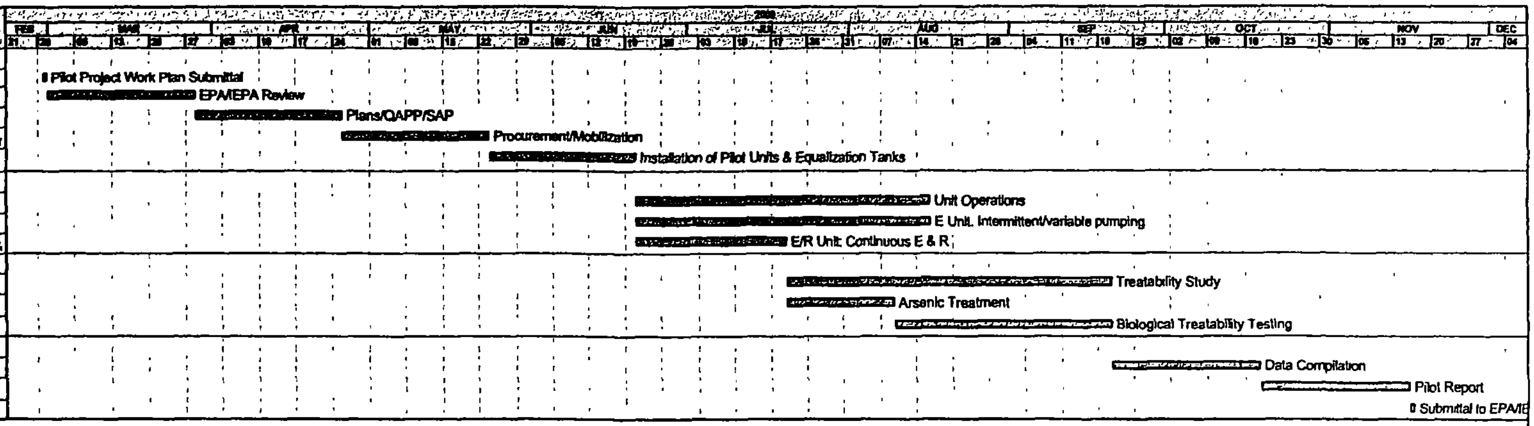


Figure 5.3 Multi-Depth Monitoring Well Nest in E Unit

Orig Dur	Rem Dur	Early Start	Early Finish	Description
1	1	29FEB00	29FEB00	Pilot Project Work Plan
20	20	01MAR00	28MAR00	EPA/IEPA Review
20	20	29MAR00	25APR00	Plans/QAPP/SAP
20	20	26APR00	23MAY00	Procurement/Mobilizatior
20	20	24MAY00	20JUN00	Installation of Pilot Units
40	40	21JUN00 *	16AUG00	Unit Operations
40	40	21JUN00	16AUG00	E Unit:
20	20	21JUN00	19JUL00	E/R Unit: Continuous E &
45	45	20JUL00	20SEP00	Treatability Study
15	15	20JUL00 *	09AUG00	Arsenic Treatment
30	30	10AUG00 *	20SEP00	Biological Treatability
20	20	21SEP00	18OCT00	Data Compilation
20	20	19OCT00	15NOV00	Pilot Report
1	1	16NOV00	16NOV00	Submittal to EPA/IEPA



Start date	29FEB00
Finish date	16NOV00
Data date	29FEB00
Run date	23FEB00
Page number	1A

Figure 9.1
Waukegan Pilot Project Work Plan

APPENDIX B

FIELD AND LABORATORY STANDARD OPERATING PROCEDURES

	Title	*SOP Number*
A.	Field SOPs	
	1. pH/Temperature	PHT-15670
	2. Conductivity	SC-15670
	3. Turbidity	NTU-15670
	4. Dissolved Oxygen	DO-15670
	5. Oxidation-Reduction Potential	ORP-15670
B.	Laboratory SOPs	
	1. Cooler Receipt	REC-5
	2. Sample Receipt and Log-in	REC-7
	3. VOCs - 8260B	3-VOA-5
	4. Semivolatile Organics Extraction	3-SVO-1
	5. SVOCs - 8270C	3-SVO-37
	6. Metals Digestion	MET-3
	7. Arsenic Analysis	MET-42
	8. Alkalinity	WCM-13
	9. Ammonia Distillation	WCM-25
	10. Ammonia Analysis	WCM-58
	11. Ion Chromatography Analysis (Br, NO_3)	WCM-60
	12. Ion Chromatograph Operation	WCM-59
	13. Chemical Oxygen Demand	WCM-39
	14. Cyanide Distillation	WCM-37
	15. Cyanide Analysis	WCM-23
	16. Thiocyanate	WCM-65
	17. Total Phenolics Distillation	WCM-3
	18. Total Phenolics Analysis	WCM-8
	19. Auto-Analyzer Operation	WCM-29
	20. Total Suspended Solids	WCM-1
C.	STRATIGRAPHICS SOPs	

pH/TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 150.1

Sensitivity: 0.01 pH unit; 0.1 ^{o}C

Optimum Range: pH 1.00 to 12.00; temperature -5 to 50 ^{o}C

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated pH meter, YSI Model 3560 Water Quality Monitoring System;

2. Combination pH electrode YSI Model 3530;

3. Thermilinear thermister YSI Model 3510 temperature probe;

4. pH buffer solutions, pH 4.00, 7.00, and 10.00 (certified buffer solutions);

5. Distilled or deionized water in wash bottle.

Calibration:

1. Switch On/Off key to On. Before connecting the pH electrode, zero the electronics with the shorting cap attached to the meter. Turn on the meter and set the pH function switch to pH. Connect the shorting cap to the pH input jack and set the manual temperature compensation knob to 25°C. Adjust the CAL control to indicate 7.00 ±0.01 on the pH-mV display. Disconnect the shorting cap from the pH input and connect it to the mV input jack. The monitor is now zeroed.

2. Test the 3530 pH electrode for noise and offset as follows: Rinse the 3530 and the YSI 3510 Temperature Probe with pH 7.00 buffer to remove any contaminants. Connect the 3530 to the pH input jack and the 3510 to the TEMP input jack. Pour pH 7.00 buffer into a 50 mL sample cup then immerse both of the sensors into the buffer at 25.0 ±0.1°C (use the °C display to confirm the temperature). Allow the sensors to equilibrate. A display value other than 7.00 shows electrode background noise and offset. The 3530 background noise and offset at pH 7.00 should not exceed ±0.2 pH units at 25°C. Replace pH probe if background noise exceeds this tolerance.

3. Set the function switch to pH ATC. Connect the 3510 to the pH ATC input jack. While the 3510 can be used in ether location, the pH ATC function will not work unless the 3510 is connected to the pH ATC input.

4. Rinse the 3530 and a YSI 3510 Temperature Probe with pH 7.00 buffer to remove any contaminants. Connect the 3530 to the pH input jack and the 3510 to the TEMP input jack. Pour pH 7.00 buffer into a 50 ml sample cup, immerse both of the sensor into the buffer. Allow the sensors the equilibrate in the buffer until a stable reading is obtained. Read the temperature and adjust the pH manual temperature compensation knob to the same value. Adjust the CAL control knob for 700 ±0.01 pH units in the display and discard the buffer. Rinse the sensors with deionized or distilled water, followed by a rinse of the next desired buffer (typically pH 4.00 or 10.00). Half fill another disposable 50 ml sample cup with the next buffer for calibration and immerse the sensors. Allow the sensors to equilibrate until a stable reading is obtained. The temperature of the two buffers should not differ by more than ±0.1°C. Adjust the SLOPE control until the display is within 0.01 pH units of the buffer;s stated value. Discard the buffers. The pH system is now calibrated and ready for use.

Procedure:

1. Calibrate meter using calibration procedure.
2. Set up meter as outlined in the operating maual.
3. Pour the sample into clean sample jar or plastic cup.
4. Record temperature and pH of the sample in the logbook.
5. Rinse with water and pH 7.00 buffer.
6. Repeat steps 3 through 5 for each sample.
7. Recheck calibration with pH 7.00 buffer solution after every 10 or fewer samples and after the last sample.
8. Store pH electrode in soaker bottle when not in use.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ±0.2 pH units.

   If the results are outside of the control limits, rinse electrodes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If drift is suspected to be the cause of the problem, clean the electrode and recalibrate. If drift is still apparent, replace electrode.

2. Calibration check results must be ±0.10 pH unit of the true value. If the result is outside of ±0.10 pH unit, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

3. All glassware is to be soap and water washed, tap water rinsed and distilled or deionized water rinsed prior to analyses.

Interferences:

Interferences in pH measurements occur with presence of weak organic and inorganic salts and oil and grease. If oil and grease are visible, note in logbook. Clean electrode with soap and water, followed by 10% HCl and deionized water rinse. Recalibrate meter before analysis of next sample.

CONDUCTIVITY

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance

Reference: "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, revised March 1983, Method 120.1

Sensitivity: 0.1 mmhos/cm

Optimum Range: 0 - 100.0 mmhos/cm

Sample Handling; Determine on site

Reagents and Apparatus:

1. Conductivity meter - YSI Model 3560 Water Quality Monitoring System;
2. Conductivity Cell - YSI Model 3520 Flow-Through Conductivity Cell (K=5/cm);
3. Thermilinear Thermister - YSI Model 3510 Temperature Probe;
4. Deionized water;
5. Conductivity standard, 1.0mmho/cm @25°C - YSI Model 3167.

Notes:

The conductivity meter is factory calibrated. The calibration is checked using a solution of known conductance.

Calibration Check

Connect the 3520 cell and a 3510 Temperature Probe to the 3500, and remove them from the sample chamber. Set the conductivity function switch to 2 ATC. Rinse the inside and outside of the cell and the probe with about 1/3 the content of the 3167 bottle. Place both of the sensors into the remainder of the solution in the bottle and allow them to come to temperature equilibrium. Make sure that the 3250 body is immersed so that the liquid level is half way up the knurled portion of the cell. Read the displayed value and determine if the cell/instrument is within specified accuracy. The displayed value is corrected to 25°C automatically and should be 1.000 ±070 mmho/cm. If the value is not within specification replace 3250 cell.

Procedure:

1. Check calibration of meter.

2. Set up meter as outlined in the operating manual.

3. Before any conductivity cell is used, it should be soaked in distilled or deionized water for at least one hour. To make conductivity measurements, connect a YSI 3520 Flow-Through Conductivity Cell to the 3500. Set the conductivity function switch to 2 and observe the displayed value after the reading is stable. The display reads out in mmho/cm.

4. If the overrange signal (1.________) is displayed, the conductivity of the water being measured is greater than 1.999 mmho/cm. Reset the function switch to 20. If the overrange signal is still displayed, reset to 100. If the overrange signal is still displayed, either the conductivity is greater than 100.0 mmho/cm and the YSI 3500 Water Quality Monitor can not be used for conductivity determinations.

5. Record conductance readings in field logbook.

6. Repeat steps 3 through 5 for remaining samples.

Quality Control:

1. The quality control calibration check standard must be analyzed initially, after every 10 or fewer samples and after the last sample. If less than 10 samples are analyzed, the calibration standard is still required to be analyzed. The standard must be within ±10 percent of the true value or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standard in the field logbook.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicate values are to be within ±15% of each other. If outside of this range, reanalyze the samples. If still outside the acceptance range, recollect sample and reanalyze. If still out, replace probe.

TURBIDITY

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Nephalometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 180.1

Sensitivity: 0.01 Nephalometric Turbidity Unit (NTU)

Optimum Range: 0 - 20; 0 - 200 NTU

Sample Handling: Determined on site

Reagents and Apparatus:

1. Direct reading turbidity meter, HF Scientific Model DRT-15C;
2. Cuvettes with screw tops;
3. Battery charger;
4. 0.02 NTU (nominal) reference standard;
5. Distilled or deionized water in wash bottle.

Calibration Check and Operation

The turbidimeter has been calibrated by the manufacturer and electronic calibration using freshly prepared formazin standards should only be performed if the electronic printed circuit board, the photodetectors or the light source has been replaced. The calibration procedure is presented in pages 5 and 6 of the operating manual (attached).

The procedures for calibration checks and the operation of the meter follows:

1. For accurate measurements in the low range rotate the cuvettes in the well to obtain the minimum reading. Mark the cuvette with one of the adhesive dots provided with the instrument so that orientation of the cuvette will be identical each time it is placed in the instrument.

2. To operate the turbidimeter, switch to the "20" range and place the Reference Standard (0.02 NTU) in the optical well.

3. With the light shield in place over the well, adjust the Reference Adjust knob to cause the meter to read the reference standard value on the scale. The unit is now ready for use in either range.

4. To make a measurement of a sample, clean one of the cuvettes and fill to within approximately 1/2" of top with sample. Place the top on the cuvette and carefully clean the outside surface of the cuvette with a lint free wiper such as KimWipes. Place the sample in the well and place the light shield over the well. Select the appropriate range for best readability. Record results in field logbook.

5. Repeat steps 3 and 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within ±15%.

   If the results are outside of the control limits, clean cuvettes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below).

2. Calibration check results must be ±10% of the true value. If the result is outside of ±10%, clean cuvettes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

3. All glassware is to be soap and water washed, tap rinsed and distilled or deionized water rinsed prior to analyses.

Interferences:

Interferences in turbidity measurements are generally due to dirty or scratched cuvettes. Handle only the top one-third of the cuvettes and wipe clean using a lint-free wiper (KimWipes or equivalent).

DISSOLVED OXYGEN

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 360.1

Sensitivity: 0.1 mg/L as O_2

Optimum Range: 0.1 mg/L to 20 mg/L O_2

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated dissolved oxygen (DO) meter, Corning Check Mate System;
2. Zero oxygen standard;
3. DO sensor filling solution;
4. DO membrane replacement kit;
5. Distilled or deionized water in wash bottle.

Setting Up DO Sensor:

The sensor is shipped dry and must be filled before use.

1. Unscrew the membrane cap from sensor and fill using DO electrolyte.
2. Tap membrane cap gently to remove air bubbles. Gently screw cap onto probe body allowing surplus electrolyte to run out. (Caution: Do not overtighten)
3. Fit sensor to meter module.
4. Allow 30 minutes for polarization of electrode.

Calibration:

1. Remove wetting cap from tip of sensor. Switch on meter.

2. For first calibration point, place sensor in zero oxygen solution. Allow sufficient time for sensor to stabilize.

3. Move the sensor in a gentle circular motion.

4. Make sure sensor is immersed to a depth of 40 mm to cover the temperature sensing element.

5. Press "CAL" key. CAL 1 is displayed on meter and after endpointing, the display automatically updates to zero.

6. For second calibration point, hold sensor in air. Press "CAL" key. CAL 2 is displayed. After endpointing, the display automatically updates to 100% O_2.

7. To adjust oxygen calibration for salinity and barometric pressure, press "Mode" key. In mg/L O_2 mode, press "CAL" key and 100 is displayed. Use down arrow and up arrow on the keypad to adjust the display according to the salinity and barometric pressure tables contained on the operating instruction leaflet.

Procedure:

1. Calibrate meter using calibration procedure.

2. Pour the sample into clean sample jar or plastic cup.

3. Place sensor in sample. After following the immersion, stirring and stabilization steps referred to during calibration, press "READ" key to obtain sample result.

4. Record result in the field logbook.

5. Repeat steps 2 through 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within 15%.

   If the results are outside of the control limits, rinse electrode and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If unable to recalibrate, replace sensor membrane.

2. Calibration check results must be within 10% of the true value. If the result is outside of 10%, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in control calibration.

3. All glassware is to be soap and water washed, tap rinsed and distilled or deionized water rinsed prior to analyses unless pre-cleaned sample jars are used.

Interferences:

Interferences in DO measurements generally occur due to membrane coating. Clean probe as specified in the sensor manual.

The presence of other gases such as chlorine, nitrous and nitric oxide, hydrogen sulfide and sulfur dioxide interfere with DO measurements. The sulfur based compounds will tarnish the electrodes resulting in sluggish or erratic measurements. Polishing the electrodes as specified in the operating manual will restore the performance of the meter. Recalibrate meter before analysis of next sample.

OXIDATION-REDUCTION POTENTIAL (ORP)

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Standard Methods for the Examination of Water and Wastewater", APHA, 18th edition, 1992, Method 2580B.

Sensitivity: 1 mV

Optimum Range: -1,500 to 1,500 mV

Sample Handling: Determined on site

Reagents and Apparatus:

1. ORP meter, YSI Model 3560 Water Quality Monitor;
2. ORP electrode assembly, YSI Model 3540;
3. Thermilinear thermistor temperature probe, YSI Model 3510;
4. ZoBell Solution, YSI Model 3682;
5. Distilled or deionized water in wash bottle.

Calibration:

1. Turn on the YSI 3500 Water Quality Monitor and set the pH function switch to mV.
2. Connect the shorting cap attached to the 3500 to the mV input jack. The display should read 000 ±2 mV. This indicates that the 3500 electronics are zeroed.
3. Detach the shorting cap and connect the 3540 to the mV input jack. If a pH electrode is not attached to the pH input jack, connect the shorting cap to it.
4. Attach the 3510 to the TEMP input jack.
5. Rinse the 3540 and 3510 with distilled or deionized water, followed by a rinse with a small amount of reconstituted YSI 3682 ZoBell Solution.

6. Half fill a disposable 50 ml sample cup with ZoBell Solution and fully immerse the bulb of the 3540 and the end of the sheath of the 3510. Allow the sensors to equilibrate, and note the reading.

7. The displayed mV values is not temperature compensated and should be corrected to 25°C at 1.3 mV/°C. The temperature coefficient is in reverse proportion to the temperature.

8. Correct the value to 25°C using the following equation:

   Actual Value mV = Display Value + [(Display Temp. - 25°C) x (1.3 mV)]

Procedure:

1. Calibrate meter using calibration procedure.
2. Set up meter as outlined in the instruction manual.
3. Record temperature and ORP of the sample in the field logbook.
4. Correct ORP to 25°C using the formula presented above.
5. Record corrected ORP in the field logbook.
6. Repeat steps 3 through 6 for each sample.
7. Recheck calibration with ZoBell solution after every ten or fewer samples and after the last sample.
8. Store electrode in soaker bottle when not in use.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ±10 mV.

   If the results are outside of the control limits, rinse electrodes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If drift is suspected to be the cause of the problem, clean the electrode and recalibrate. If drift is still apparent, replace electrode.

2. Calibration check results must be 231±10 mV. If the result is outside of this range, rinse electrodes and check solution again. If still outside the this range, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

Interferences:

Interferences in ORP measurements occur when the platinum electrode surface becomes coated. Clean the ORP electrode as follows:

1. Soft coatings should be removed by use of a wash bottle of water or by gently wiping with a soft cloth. Remove the bulb guard if necessary. Be careful not to scratch the platinum.

2. Hard coatings or organic chemicals should be removed by an appropriate chemical solvent, by gently scrubbing with a very fine cleansing powder such as "Softscrub", or by gently polishing with 600 grade wet silicon carbide paper. Wet a piece of the paper with water and gently polish the electrode with a circular twisting motion.

Note:

After cleaning the platinum surface, soak the electrode for a 8 to 24 hours in 4.0 pH buffer, then recheck it with YSI 3682 ZoBell Solution before further use.

STANDARD OPERATING PROCEDURE

TITLE: Cooler Receipt Log

APPLICATION: Sample Receiving.

PROCEDURE:

A Cooler Receipt Log is completed for each shipment of coolers from a project to document that appropriate items have been checked during cooler receipt and log-in, and also, that appropriate notifications have been made to laboratory groups. The completed document is to become a permanent part of the workorder packet for the batch. An example Cooler Receipt Log is attached to this SOP.

The entry of samples into the laboratory workload is generally accomplished in two phases. These phases are the Receipt Phase, and the Log-in Phase. Although these two phases may be performed by one individual in one continuos action, there may also be a time delay or hand-off to another individual between the two phases. For this reason, they are broken down into two distinct sections on the Cooler Receipt Log with each phase requiring Initials and Date to be entered onto the log.

The following shall be completed and entered onto the Cooler Receipt Log as necessary:

Receipt Phase

1. Enter the Number of coolers in the shipment, and the Project Name or identification onto the log. The Batch Number will be entered during the log-in phase.

2. Record the Date that the cooler(s) were originally opened by the laboratory and the initials of the laboratory staff performing this phase.

3. Complete Steps 1 through 9 on the log, circling the appropriate response.

4. Initiate a nonconformance memo for any items necessary. For any cooler with a temperature nonconformance it is critical that all effected sample points and containers can be identified. Make a photocopy of the COC document and circle all effected samples and containers. Attach this annotated copy of the COC to the nonconformance memo for routing, with the workorder packet, to the project manager for review.

5. Make any notifications to laboratory staff which are required for items 8 and 9.

6. If the Log-in phase is to be performed at a later time store the samples appropriately, otherwise proceed to the log-in phase.

Log in Phase

1. Record the Date and the initials of the Laboratory staff performing this phase in the space provided.

2. Complete steps 1 through 11 on the log.

3. Initiate a nonconformance memo for any items necessary.

4. Once a Laboratory Batch Number is assigned, enter the number onto the log in the space provided in the upper left hand corner.

5. When completed, the log should be included in the Chain of Custody/workorder packet.

Review

The completed log should be checked for the following: completeness, that necessary contacts have been made, and that any nonconformance issues have been documented. This check should be performed by the Group leader or another trained staff member prior to the packet leaving the sample receiving area.

REVIEWED BY: Gregory J. Graf — 12-29-99
Gregory J. Graf
Quality Assurance Officer
Date

APPROVED BY: Jeffrey A. Gordon — 12-29-99
Jeffrey A. Gordon
Technical Director - Inorganics
Date

Glen A. Coder — 12-30-99
Glen A. Coder
Laboratory Manager
Date

En Chem, Inc. COOLER RECEIPT LOG

P-tch No.________________

Quality Assurance Document

roject Name or ID________________ No. of Coolers:________ Temps:________________

A. Receipt Phase: Date cooler was opened:________________ By:________________

1: Were samples received on ice? (Must be ≤ 6 C)........YES NO[2]

2. Was there a Temp. Blank?........YES NO

3: Were custody seals present? (Record on COC)........YES NO

4: Are COC documents present?........YES NO[2]

5: Is this Project a Quick Turn Project?........YES NO

6: Is there any sub-work?........YES NO

7: Are there any short holdtime tests?........YES NO

8: Are any samples nearing expiration of hold-time? (Within 2 days)........ YES[1] NO Contacted by/Who________________

9: Do any samples need to be Filtered or Preserved in the lab?........ YES[1] NO Contacted by/Who________________

B. Log-in Phase: Date samples were logged-in:________________ By:________________

1: Were all sample containers listed on the COC received? ....YES NO[2] NA

2: Sign the COC as received by En Chem. Completed........YES NO

ɔ sample labels match the COC? ....YES NO[2]

: Check sample pH of preserved samples. (not VOCs) Completed........YES NO NA

5: Are sample volumes adequate for tests requested? ....YES NO[2]

6: Are VOC samples free of bubbles >6mm ....YES NO[2] NA

7: Enter samples into master logbook. Completed........YES NO

8: Place laboratory sample number on all containers. Completed........YES NO

9: Complete LTS sheet. Completed........YES NO

10: Complete nonconformance record if applicable. Completed........YES NO NA

11: Initiate Subcontracting procedure, SOP 1-REC-4, if applicable. Completed........YES NO NA

Short Hold-time tests:

48 Hours or less	7 days	Footnotes
Coliform (6 hrs)	Flashpoint	1 Notify proper lab group immediately.
Hexavalent Chromium (24 Hrs)	TSS	2 Complete nonconformance memo.
BOD	Total Solids	
Nitrite	TDS	
Ortho Phosphorus	Sulfide	
Turbidity	Free Liquids	
Surfactants	Total Volatile Solids	
Sulfite	Aqueous Extractable Organics- ALL	
En Core Preservation	Unpreserved VOC's	
ʼr	Ash	

ʾev. 10/20/99, Attachment to 1-REC-5.
ʖubject to QA Audit. Reviewed by/date________________

g:/data/qcdept/lobdocs/reccrl.doc Controlled copy has red header.

Standard Operating Procedure

Title: Sample Receipt and Log-in

Department: Receiving

Procedure:

1. Receiving Access and Custody

1.1. Access to the laboratory is controlled. The front entrance is open between 8 am and 5 pm. Except for the front entrance, outside doors to the laboratory are kept locked. Visitors sign a visitor's log by the front entrance and are escorted by En Chem personnel while in the laboratory. All laboratory personnel are issued a security card that permits entry to the laboratory during off hours.

1.2. Samples are either received directly from the client, via En Chem's couriers, or from commercial courier services such as UPS, Federal Express, Airborne Express, and others. Only packages addressed to En Chem are received.

1.3. Custody of samples between the sampling event and En Chem is maintained through a Chain of Custody (COC) which is a written record of sample bottle possession and transfer (see SOP REC-8). This form includes client information, project information, sample descriptions, field ID's, sample collection dates and times, analyses requested, filtering information, bottle preservation codes, matrix type, and a record of when and by whom the samples were relinquished and received. If any items received by the laboratory are not in conformance with what the client wrote on the COC, complete a Sample Entry Nonconformance Memo (see SOP REC-5).

2. Sample Receipt :Several steps are taken immediately upon receipt of the package.

2.1. In a fume hood, open the package or cooler and remove the paperwork.

2.1.1. Initiate a Cooler Receipt Log per En Chem SOP REC-5.

2.1.2. Use the thermometers located in Sample Entry to take the temperature of the temperature blank bottle included with the shipment. Record this temperature on the COC in the shaded area.

2.1.3. The requirement for cooling is less than 6°C. If the temperature reading is higher than 6°C, complete a nonconformance memo documenting the problem.

2.1.4. If there is no temperature blank, then take the temperature of an unpreserved water sample located near the center of the cooler using the IR Temperature gun.

2.2. Remove the shipping airbill/ tracking number from the outside of the package and affix it to a plain white sheet of paper.

2.2.1. Clip this paper to the COC.

2.2.2. Mark the project name on the package.

2.3. Officially take custody of the package by signing the COC.

2.3.1. If the En Chem courier or the client delivered the samples, have them sign the COC in the space marked "Relinquished By."

2.3.2. If the samples came via a commercial courier service, sign the COC for the courier including the date and time it was delivered under "Relinquished By."

2.3.3. Sign your name and the time and date of sample receipt in the "Received By" space on the COC.

2.3.4. Any packages received after hours will be placed in the walk-in cooler and must be logged in first thing the following day.

2.4. Check for a Scope form, which is filed alphabetically by project name in the cabinet located in Sample Entry.

2.4.1. If one is present, attach it to the COC.

2.4.2. If a scope is not present or if a problem exists between the scope and the COC, notify customer support.

3. Sample Log-in

3.1. Beginning with projects which have a quick turn-around time or samples with a short hold time, unpack the samples and arrange them on the receiving counter in the order given on the COC.

3.2. Verify the cooler contents against what is written on the COC.

3.2.1. Record any discrepancies in the Total Bottles section located in the shaded area of the COC and document in a nonconformance memo.

3.2.2. Record the total number of bottles received for each station ID in the Total Bottles section.

3.3. Note if any samples are in poor condition (e.g. broken bottles, cracked caps, mislabeling, wrong container type for analysis to be run, etc.). Record this information in the Total Bottles section of the COC for each affected station ID and document in a nonconformance memo.

3.3.1. Replace any cracked caps with new ones. Place an "x" on the caps that are replacements. Note in the comments section of the COC whether it appears that any sample has leaked out or the meltwater has leaked in the sample.

3.4. Check all water volatile samples and non-Wisconsin soil volatile samples for headspace.

3.4.1. Tap VOC water sample vials lightly and stand water samples on end to see if there are any air bubbles in the vial. Water samples may not have bubbles which exceed 6 mm in diameter.

3.4.1.1. If bubbles exceeding 6 mm are present in all the volatile bottles for a given station ID, write "Headspace" in the Comments section of the COC and document in a nonconformance memo. If in doubt, notify a volatile organics staff member for determination.

3.4.2. Soil samples should be in appropriate bottles (60 ml or 125 ml jars with teflon-lined caps) with as little space in them as possible. Document conditions other than these (i.e. jar half filled with coring sample) in a nonconformance memo.

3.5. If a set of trip blanks was sent for a project containing samples for volatile analysis, and if the sampler has not already done so, write "Trip Blank" in the next available station ID space on the COC, initial and date the entry, and record the total number of trip blank vials received.

3.6. If a set of trip blanks was not sent for a project containing samples for volatile analysis, "No trip blank received" will be recorded in the total bottles section on the COC and documented in a nonconformance memo.

3.7. For any projects specifically require a storage blank, prepare the blank as follows.

3.7.1. Fill a HCl preserved 40 ml volatile vial with Milli-Q water (organic free) and label it "Storage Blank". Date and initial it.

3.7.2. Document the storage blank in the next available station ID line on the COC and during the numbering process (see Section 3.11) give it a laboratory number.

3.8. Except for those samples requiring no headspace (i.e. volatile samples), check the pH of any preserved samples with pH indicator paper. This activity should be performed in a hood.

3.8.1. The pH of preserved samples is checked, using wide range pH paper, by dipping the paper into the preserved container. If requested by a client, pH can be checked by decanting a small amount of sample into a disposable plastic cup and then dipping the paper into the cup. The sample aliquat used should be discarded following pH determination.

3.8.2. If the pH of an acid preserved sample is greater than 2, add the appropriate preservative to the sample until the pH is less than or equal to 2. Some projects may require approval by the client before laboratory preservation, i.e. US Army Corps. If approval is required, place the sample on hold and notify the project manager. Upon approval add the appropriate preservative to the sample until the pH is less than or equal to 2.

3.8.2.1. Use the same preservatives that are used in the bottle preparation room. Record the reagent code for the preservative used in a nonconformance memo if requested by the client.

3.8.2.2. Document the issue, resolution, and volume of preservative added to the sample in a nonconformance memo.

3.8.2.3. Write "Adjusted" under Sample Receipt pH on the COC.

3.8.3. If the pH of a sample preserved with a base is less than 12, add the appropriate preservative to the sample until the pH is greater than or equal to 12. Some projects may require approval by the client before laboratory preservation, i.e. US Army Corps. If approval is required, place the sample on hold and notify the project manager. Upon approval add the appropriate preservative to the sample and document as in 3.8.2.1 thru 3.8.2.3.

3.8.4. If none of the samples require pH adjusting, write "Acceptable" or "OK" under Sample Receipt pH on the COC.

3.9. Check the collection dates and times to make sure that the requested analyses are still within their hold times. Use the Sampling and Sample Preservation List in the three-ring binder located in receiving.

3.9.1. Contact the appropriate analyst about any samples with 48 hours or less remaining on hold time.

3.9.2. If a sample's hold time has expired, document this in a nonconformance memo.

3.10. Check to make sure there is enough volume for each station ID.

3.10.1. Check the number of bottles, the preservation, and the volume for the analyses listed on the COC. Use the volumes given on the Sampling and Sample Preservation List.

3.10.2. If there is not enough volume to perform all of the requested analyses, contact the group leader of the lab which does that analysis or customer services to see if the analyses can still be performed and/or what analyses the client wishes to prioritize. Record any volume issues in a nonconformance memo.

3.11. If there are any samples with a quick turn-around time, notify the appropriate section supervisor, analyst, or technician. Provide a copy of the COC document at that time.

3.12. Assign the laboratory numbers to the samples for a project receipt.

3.12.1. Assign the next consecutive Batch Number in the master log-in book to a project receipt.

3.12.2. Document the date of receipt and log-in, the project name and number, the matrix "type", and the Batch Number in the next available space in the master log-in book.

3.12.3. Write the Batch Number on the top of the COC laboratory number column or on the 1st line of that column.

3.12.4. Assign each sample a Laboratory Information Management System (LIMS) Number, sequentially numbering each Sample Description / Field ID on the COC as -001, -002, -003, etc. in the order listed on the COC in the space designated "Laboratory Number".

Note: Be sure that samples of different matrices with the same field ID are given separate LIMS numbers.

3.13. Record the bottle types, bottle volumes, type of preservation used, and samples storage location in the master log-in book.

3.13.1. If the complement of bottles included for each Sample Description / Field ID is the same as the bottles for another Sample Description / Field ID in that project, the bottle types, etc. can be recorded on one line in the log-in book.

3.13.2. If the bottle types from a sample are different from those for other samples, record those on a separate line in the log book. Use additional spaces in the log-in book as necessary.

3.13.3. There is a section for each type of preservation in each line in the log-in book. Within each section, there is a square for each of the possible bottle volumes for that preservation. Record the total number of bottles for each bottle volume and preservation type in these squares on each line.

3.13.4. Some of the squares in the manual log-in book are split diagonally into two triangles. Any plastic bottles must be entered in the top triangle, while glass bottles are entered in the bottom triangle.

3.13.5. Use the location codes listed on the bottom of each page of the log-in book to document where each bottle type will be stored.

3.13.5.1.Samples that are preserved with H_2SO_4, unpreserved samples, and soil samples must be refrigerated and put in the walk-in cooler.

3.13.5.2.Volatile organic samples must be stored in the volatile refrigerator located in the volatile analysis lab. Any samples with an odor or that are known to be high level should be placed into the small refrigerator in the VOA lab to segregate them from other samples.

3.13.5.3.Samples preserved with HNO_3 do not need refrigeration and can be stored in storage area "A".

3.13.6. The number of bottle types for each station ID must be entered in the log book with the location code preceding the total number of bottles for each square or triangle. For example, entries such as V-1, W-2, and A-3 mean 1 bottle is located in the volatile refrigerator, 2 bottles are located in the walk-in cooler, and 3 bottles are located in the archive area.

3.14. Initial the entry in the log book.

3.15. The COC Total Bottles section for each entry is for recording any unusual aspects (e.g. bottle types, labeling issues etc.) and number of bottles received.

3.16. Numbering the samples.

3.16.1. Line up the sample bottles by Sample Description / Field ID, the same way as listed on the COC, before numbering the samples.

3.16.2. Generate the labels and attach them to the appropriate sample bottles. Take care to associate the proper bottle with the proper sample number using the sample point description on the COC and the bottle label.

3.17. QC The Samples: A person not involved with the numbering process verifies the correct numbering / labeling of the sample bottles (matches the COC).

3.17.1. Initial the appropriate Batch Number section of the log-in book to indicate that the samples have been QC'd.

3.18. Seal all volatile analyses sample bottles in zip-lock bags.

3.18.1. Place each sample point (lab #) in its own bag separate from other samples.

3.18.2. Place the samples for a given WO# together in a plastic bin.

3.18.3. Label the bin with the project name, the work order number, and the number of samples.

3.19. Put the samples into their respective storage locations.

3.19.1. Write up a Location Tracking System (LTS) sheet for each location that the samples are to be stored.

3.19.2. For each LTS / location, record the bottle types, the sample number range of those bottle types, the project name, the project number, the matrix type, preservative, the date logged in, and your initials on the LTS sheet.

3.19.3. As you put the samples into their respective locations, record the shelf ID of their location on the LTS sheet.

3.19.4. Samples can be moved anywhere on the shelf it was logged into but cannot be moved from shelf to shelf.

3.19.5. Place the white copy of the LTS sheet into the three-ring binder for that storage location in order by batch number.

3.19.6. Attach the yellow copy of the LTS sheet to the COC.

3.20. Copy the COC documents to all laboratory departments for which samples were received. Place these copies into the labeled bins in the sample receiving area. The supervisors check these periodically throughout the day.

3.21. Assemble all paperwork into a work order packet. At a minimum, the completed WO packet consists of several items. (Forms are shown in Appendix 1 of this SOP as example. The use of these forms is addressed in other SOP's.)

3.21.1. The COC.

3.21.2. Cooler Receipt Log.

3.21.3. Any Nonconformance Memos.

3.21.4. The Scope form, if available.

3.21.5. The airbill or tracking number from the package.

3.21.6. The yellow copy from all LTS sheets.

3.21.7. A copy of any subcontracting COC paperwork.

3.21.8. Any other documentation associated with the project or receipt of these samples.

3.22. Put the work order packet into the LIM's entry bin.

4.0. The Batch Number for the project is then created in LIM's and the packet is routed to the project manager for review prior to LIM's entry.

Approved By: ______________________ 3/7/2000
Renee Breed — Date
Group Leader

Approved By: ______________________ 3-7-2000
Gregory J. Graf — Date
Quality Assurance Officer

Approved By: ______________________ 3-7-2000
Jeffery A. Gordon — Date
Technical Director

Approved By: ______________________ 3-7-2000
Glen A. Coder — Date
Laboratory Manager

(Please Print Legibly)

Company Name: ______

Branch or Location: ______

Project Contact: ______

Telephone: ______

Project Number: ______

Project Name: ______

Project State: ______

Sampled By (Print): ______

EN CHEM INC.

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CHAIN OF CUSTODY

45807

*Preservation Codes

A=None B=HCL C=H2SO4 D=HNO3 E=EnCore F=Methanol G=NaOH

FILTERED? (YES/NO)

PRESERVATION (CODE)*

ANALYSES REQUESTED

Page ____ of ____

P.O. # ______ Quote # ______

Mail Report To: ______

Company: ______

Address: ______

Invoice To: ______

Company: ______

Address: ______

Mail Invoice To: ______

Data Package Options (please circle if requested)

QC Summary	Surcharge	Site-Specific QC Required?
EnChem Level II	Std. Delivery	Yes No
EnChem Level III	10% (min. $50)	(If yes, indicate QC sample and submit triplicate volume)
EnChem Level IV	25% (min. $100)	

LABORATORY ID (Lab Use Only)	[illegible]	[illegible]	[illegible]											MATRIX	COMMENTS	TOTAL BOTTLES (Lab Use Only)

Turnaround Time Requested (TAT) (circle): Std (10 Bus. Days) Rush

(Rush TAT subject to approval/surcharge)

Quick Turn Number: ______

Date Needed: ______

Transmit Rush Results by (circle): Phone Fax

Phone #: ______

Fax #: ______

Samples on HOLD are subject to special pricing and release of liability

TAT	Std. Surcharge
1 day	3.0x
2 day	2.0x
3 day	1.5x
4 day	1.4x
5 day	1.3x

Matrix Codes

W=Water

S=Soil

A=Air

C=Charcoal

B=Biota

SL=Sludge

Relinquished By:	Date/Time:	Received By:	Date/Time:	
Relinquished By:	Date/Time:	Received By:	Date/Time:	En Chem Project No.
Relinquished By:	Date/Time:	Received By:	Date/Time:	Sample Receipt Temp.
Relinquished By:	Date/Time:	Received By:	Date/Time:	Sample Receipt pH (Wet/metals)
Relinquished By:	Date/Time:	Received By:	Date/Time:	Custody Seal
Relinquished By:	Date/Time:	Received By:	Date/Time:	

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Madison, WI 53711
608-232-3300 • Fax: 608-233-0502
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EN CHEM INC.

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1-800-7-ENCHEM

EN CHEM FIELD BOTTLE REQUEST FORM

Shipping Charge:____________

Client:______________________________
Project #:______________________________
Project Name:__________________________
SampleDate:___________________________
SampleType:___________________________

Date Bottles Needed:______________
Bottle Requestor:________________
Date Requested:_________________

Bottle Preparer:________________
Date Prepared:_________________
Checked By:____________________

1) Is this a Wisconsin regulated project? Y N
2) Is there a residual chlorine in the water samples? Y N

Sample Points:

Parameters:

# of Bot.	Size	Type	Preserv.	Associated Parameters	Bottle Lot Number

Deliver to: ______________________________
____
____
____

Method of Shipment:______________

Number of coolers: ________

Controlled copy has red header.

En Chem, Inc.

Quality Assurance Document

Master Logbook Form

Date	Project Information				Biota	HNO3 (pl/gl)					H2SO4 (pl/gl)				Unpreserved (pl/gl)							HCL		MeOH	NaOH		ZnAc+ NaOH	Log'd by
Yr___	Client Information	Matrix	Batch #	Sample #		125	250	500	1L	4L	125	250	500	1L	encore	40	60	125	250	500	1L	40	1L	60	250	500	1L	
	Submitter Name																											
	Project Name																											
	Project #																											
	Comments.																											QC
	Submitter Name																											
	Project Name																											
	Project #																											
	Comments:																											QC
	Submitter Name																											
	Project Name																											
	Project #																											
	Comments:																											QC
	Submitter Name																											
	Project Name																											
	Project #																											
	Comments.																											QC

Locations

A Archive Room
D&E Semi-volatiles (water)
F Semi-volatiles (soil)
G Semi-volatiles (extracts)
S Subcontracted after receipt

V Volatiles
W Walk-in Sample Receiving
X Walk-in Wet-Chemistry
Z Freezer

Controlled copy has red header.

LTS

Shelf	Batch & Sample Number(s)	Bottle type & Preservation	in	out	in	out	in	out	in	out	in	out	in	Depleted Y/N Date	Disposal Date & Haz Code	Return or Store

Comments:____

Storage Location__________

Disposed

Project____________________ Project #__________ Sample Type____________ Location____________

En Chem, Inc.

Quality Assurance Document

COOLER RECEIPT LOG

Batch No.____________________

Project Name or ID______________________ No. of Coolers:__________Temps:________________

A. Receipt Phase: Date cooler was opened:____________________ By:____________________

1: Were samples received on ice? (Must be ≤ 6 C)........YES NO[2]

2. Was there a Temp. Blank?........YES NO

3: Were custody seals present and intact? (Record on COC)........YES NO

4: Are COC documents present?........YES NO[2]

5: Is this Project a Quick Turn Project?........YES NO

6: Is there any sub-work?........YES NO

7: Are there any short holdtime tests?........YES NO

8: Are any samples nearing expiration of hold-time? (Within 2 days)........YES[1] NO Contacted by/Who_____

9: Do any samples need to be Filtered or Preserved in the lab?........YES[1] NO Contacted by/Who_____

B. Log-In Phase: Date samples were logged-in:____________________ By:____________________

1: Were all sample containers listed on the COC received and intact?........YES NO[2] NA

2: Sign the COC as received by En Chem. Completed........YES NO

3: Do sample labels match the COC?........YES NO[2]

4: Check sample pH of preserved samples. (not VOCs) Completed........YES NO NA

5: Are sample volumes adequate for tests requested?........YES NO[2]

6: Are VOC samples free of bubbles >6mm........YES NO[2] NA

7: Enter samples into master logbook. Completed........YES NO

8: Place laboratory sample number on all containers. Completed........YES NO

9: Complete LTS sheet. Completed........YES NO

10: Complete nonconformance record if applicable. Completed........YES NO NA

11: Initiate Subcontracting procedure, SOP 1-REC-4, if applicable. Completed........YES NO NA

Short Hold-time tests:

48 Hours or less	7 days	Footnotes
Coliform (6 hrs)	Flashpoint	1 Notify proper lab group immediately.
Hexavalent Chromium (24 Hrs)	TSS	2 Complete nonconformance memo.
BOD	Total Solids	
Nitrite	TDS	
Ortho Phosphorus	Sulfide	
Turbidity	Free Liquids	
Surfactants	Total Volatile Solids	
Sulfite	Aqueous Extractable Organics- ALL	
En Core Preservation	Unpreserved VOC's	
Color	Ash	

Rev. 2/21/2000, Attachment to 1-REC-5.
Subject to QA Audit.

Reviewed by/date__________________

g:/data/qcdept/lobdocs/reccrl.doc

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SAMPLE ENTRY NONCONFORMANCE MEMO

PROJECT NAME:			PROJECT NUMBER:	INITIATOR & DATE:	
PROBLEM	Batch Number	Station ID	Sample Entry Comments	Date/Initials	PM Comments
No Ice/Temp					
Broken Bottles/ Cracked Cap					
Hold Time					
Headspace					
Volume					
Container					
Labeling					
Preservation					
Other					

Subject to QA audit. Audited by:____________________ Date:______________

f:/data/laboomm/forms/senoncf1.xls

En Chem, Inc.

Quality Assurance Document

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. . .chemistry for the environment

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☐ Milwaukee Service Office
(414-327-5717)
☐ Central WI Sales Office
(715-693-1953)

Chain of Custody

☐ Subcontract ☐ En Chem Internal Split

From: En Chem - ________________

To: ________________

____

____

Project Due: ________________

☐ Normal Turn ☐ Quick Turn

Project State: ________________

En Chem Project No.: ________________

Requested Analysis

Filtered? Y/N

Preservative

Preservation Code

A – None B – HCL
C – H2SO4 D – HNO3
E – EnCore F – Methanol
G – NaOH O – Other

En Chem Lab No.	Client Field I.D.	Date Sampled	Sample Type	No. of Bottles								Sub Lab Sample No.

Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:

If you have questions, please contact ________________ at: ☐ Green Bay ☐ Madison ☐ Superior

Please FAX/send final report to ________________ at the: ☐ Green Bay ☐ Madison ☐ Superior Lab.

Final report to be generated by En Chem: ☐ Green Bay ☐ Madison ☐ Superior

En Chem, Inc.

Quality Assurance Document

SET No: 1

Standard Operating Procedure

TITLE: Volatile Organic Analysis by GC/MS - Methods 5030B/8260B

DEPARTMENT: Volatile Organics Laboratory

APPLICATION: The method outlined within is used for the detection of volatile organic compounds by GC/MS in waters and methanol extracted soils. See the accompanying Target Compound List (TCL) for the analytes to which this method applies.

REFERENCES: 40 CFR, Part 136, Appendix B, Revision 1.11

EPA SW-846, Methods 8260B, December 1996

EPA SW-846, Methods 5030B, December 1996

PROCEDURE SUMMARY:

Volatile compounds are purged out of water or medium level extracted solid matrices using a steady stream of helium. The compounds are trapped on a tenax/silica gel/charcoal trap, which is then heated rapidly, desorbing the compounds onto a GC/MS system. Separation occurs on a capillary column before entering the mass spectrometer, where spectra are generated that can be compared with previously prepared standard spectra. Quantitation is based upon analyte response verses standard response relative to Internal Standards.

REVIEWED BY: Gregory J. Graf (signature) — Date: 11-5-98
Gregory J. Graf
Quality Assurance Officer

APPROVED BY: Glen A. Coder (signature) — Date: 11/5/98
Glen A. Coder
Laboratory Manager

SAMPLE HANDLING AND PRESERVATION:

Water samples must be preserved with 1:1 HCl to pH <2. All samples must be stored at 4°C until analysis. Before using samples, ensure that no bubbles are found in the water samples. The sample must be flagged as having headspace if there is a bubble greater than 0.6 cm in diameter.

Samples preserved with HCl acid must be analyzed within 14 days of sampling. Samples which are not preserved must be analyzed within 7 days of sampling.

INTERFERENCES:

Interferences usually fall under two categories:

1) Masking:
This occurs when a compound is present at such a high concentration that its peak is broad enough to cover peaks with similar retention times.

2) Matrix Dampening:
This occurs when the sample, usually a soil, contains substances that impede the purging of volatiles out of the sample. This is detected by low internal standard areas and/or poor surrogate recovery.

I. APPARATUS AND MATERIALS:

HP 5970, 5971, and 5972 MSD with glass jet separator and capillary column inlet/adapter

Tekmar 2000 or 3000 Dynamic Headspace Concentrator

Archon 5100A autosampler

Dynatec PTA-30 autosampler

Capillary Gas Chromatography column - 75m x 0.53mm ID DB-624 megabore(J&W Scientific, Inc.) with a 3 µm film thickness;or, 20m X 0.18mm ID RTX-624, Restek, inc.,with a 1.0 um film thicknes.

Syringes: 5 ml Hamilton gastight with Luer-lock tip; 10, 25, 50, 100, 500, 1000 µl gastight microsyringes.

Standard solution storage containers - 1.5 ml, 7 ml, 14 ml amber vials with PTFE-lined screw caps (Pierce); 2 ml micro reaction vials with miniert valves and replaceable septa (Supelco).

REAGENTS:

Gases: Helium, UHP grade

Methanol, Burdick & Jackson Purge-and-Trap Grade or equivalent

Organic Free Water (OFW): Organic Free Water is water with organics present below MDL for most compounds. See IV. C. Method Blank Criteria.

Standards: *The following standards or the equivalents may be used,*

Primary Standard Sources:

Restek VOC Mixes (Custom and standard stock mixes)
Restek Internal Std. Mix.
Restek Surrogate Std. Mix.
Restek 4-Bromofluorobenzene Solution.

Secondary Sources:

Supelco Purgeable Kit Mixes.
Accu-standard Custom Kit Mixes.

PROCEDURE:

II. Initial Calibration

Demonstration and documentation of an acceptable initital calibration is required before any samples are analyzed. Recalibration may need to be performed as indicated by results of continuing calibration check standards.

A. Verification of Instrument Performance

A 50 ng BFB injection must meet spectral criteria as defined in SW846 8260B or CLP OLM3.1* criteria, depending on project scope requirements (see Appendix C tables 1 & 2). If the tune spectrum does not meet the criteria after several injections, then the tune file may need to be readjusted to meet the criteria before proceeding with the initial calibration.

*Note that OLM3.1 criteria is based on 3 scans and a background subtraction.

B. Preparation of Standards

Stock Reference Standards:

Stock standards may be prepared from neat standards and/or prepared mixes can be purchased. See Appendix B for the Target Compound List. A reference standard prepared from a neat standard is made up at a 10,000 ug/mL concentration as follows:

Place about 9.8 ml of methanol into a 10 ml ground-glass stoppered volumetric flask. Allow the flask to stand unstoppered for a few minutes until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1 mg. Using a 100 µl gastight syringe, add about 20-25 drops of the neat standard to the flask to obtain about 100 mg of standard. Let the drops fall just above the surface of the methanol but do not let the syringe needle touch the sides of the flask neck. Reweigh the flask, dilute to volume, stopper, then mix by inverting the flask several times. Store without headspace in a 7 mL amber vial in a freezer. All data *regarding standard preparation* must be entered into the standard preparation logbook. A number is assigned to each prepared standard solution and that number is entered on all logs where a solution is used. The vial containing a prepared standard solution must be labeled in accordance with the En Chem SOP, and include the assigned log number. A brief description of the standard must also appear on the label.

Working Standards:

Prepare working standards of both the volatile target compounds and surrogates at 100 ug/mL in P&T methanol. Prepare an Internal Standard and an Internal Standard/Surrogate solution (ISSTD) at a concentration of 250 ug/mL in P&T methanol. Record all data regarding preparation of each standard solution in the standard logbook as described above for stock standards. Label the working standard vials as described above for the stock standards. All standards should be stored in a freezer. Prepared standard solutions in methanol are stable for about 4 weeks when stored below -15°C except for volatile gas analytes, which are made on a weekly basis due to degredation. Standards should be replaced if it is determined that they have deteriorated.

Preparation of Calibration Standards:

An initial calibration consisting of at least five calibration points is analyzed before sample analysis may proceed. The standards are prepared with all target compounds, gases and surrogates at equal concentrations. The calibration levels are prepared as follows:

Calibration Level	Amount added of working standards(ul)	Volumetric
1 ug/L	1 uL	100 mL
5 ug/L	5 uL	100 mL
10 ug/L	10 uL	100 mL
20 ug/L	10 uL	50 mL
50 ug/L	25 uL	50 mL
100 ug/L	50 uL	50 mL
150 ug/L	75 uL	50 mL
200 ug/L	100 uL	50 mL

The associated volumetric is inverted three times and the contents placed into a non-preserved 40 mL VOA vial, without headspace, for instrument analysis. The autosampler adds 1uL of Internal Standard mix at 250 ug/mL into a 5mL aliquot, resulting in a final ISTD concentration of 50 ug/L. This concentration is the same for all standards, blanks and associated QC analyses.

A calibration curve for waters and medium level soil methanol extracts is purged at ambient temperature. Analyze these solutions using purge-and-trap and GC/MS methodology.

Preparation of Calibration File:

Identify the compounds using reference spectra and retention time data. Assign response factors to each compound using the internal standard technique based on its response in each standard. Refer to the formula sheet for the response factor formula in Appendix A, #1. See Appendix B for the assigned internal standard for each analyte.

Check linearity of response by calculating the percent relative standard deviation (RSD) of the response factors for each compound. The %RSD for each individual Calibration Check Compound (CCC) must be less than or equal to 30.0%. In addition, the minimum acceptable average response factor for System Performance Check Compounds (SPCC) must meet specified Response Factor (RF) critera. See Appendix C, Table 2 for lists of CCC and SPCC compounds. The validity of the calibration is accepted or rejected after making these comparisons. Some common causes of a bad calibration curve are: standard degradation, poor instrument stability or improper purge flow rate.

Any target analyte with an RSD of less than 15% is considered valid and the average response factor may be used for quantitation purposes. If the %RSD exceeds 15%, the analyst must choose the best calibration option for quantitaion purposes. Quadratic regression and third order polynomial are the other options used for analyte quantitation.

In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed within the same time clock as the calibration curve. The ICV standard must be from a second source stock and meet the same criteria as the Continuing Calibration Verification (CCV) standard before the initial calibration may be considered valid.

If the calibration is acceptable, a calibration file is constructed that matches the ID file and stores each of the five response factors, the average RF, the %RSD, and concentrations of each target analyte and surrogate.

If the calibration is not acceptable, the appropriate calibration solution(s) is reanalyzed to obtain acceptable %RSD and RFs over the entire calibration range.

III. Daily Checks and Continuing Calibration Check

A BFB tune, continuing calibration check standard, method blank, and a LCS/LCSD must be performed at the beginning of each 12 hour period during which sample analyses are performed.

A. Daily BFB Tune

To check the tune, follow the procedure in section II.A above. If the tune has failed, consider modifying the tune file again as described in section I.A above. If the BFB or the autotune fails repeatedly, source cleaning may be necessary. The BFB tune must meet the spectrum criteria as defined in Appendix C, Table 1 or 2, before proceeding with the continuing calibration check standard.

B. Continuing Calibration Check Standard

Prepare a 50 µg/L standard by adding 25 uL of the target compound working standard at 100 ug/mL into a 50 mL volumetric of organic free water. The autosampler will then add 1 uL of ISSTD mix at 250 ug/mL into a 5 mL aliquot. Compare the daily response factors with the average response factors from the initial calibration. The relative percent difference for any target analyte should not be greater than 20.0% but the percent difference for any CCC compound may not be greater than 20.0%. Some target analytes, such as the Ketones, have extremely poor purging efficiencies and may have a higher relative percent difference. If any non CCC compounds have a percent difference of greater the 20%, analyst discretion is used before sample analysis occurs. The daily response factor of any SPCC compound must be within the criteria as listed in Appendix C, Table 3. Check the validity of the ID file's qualitative matches. If the SPCC or CCC conditions are not met, the standard is not valid and should be repeated. Repeated failure to meet acceptance criteria is a sign that the initial calibration curve is no longer valid. If no extraneous causes of standard failure are suspected, the initial calibration must be repeated. Refer to IIB above.

C. Method Blank:

A method blank consists of organic free water. The method blank is analyzed in the exact manner that the samples are analyzed.

In addition to the internal standard and surrogate QA/QC requirements of sample analysis, the method blank must meet the following criteria before sample analysis may begin:

1. No common solvent (Acetone, 2-Butanone, 2-Hexanone, 4-Methyl-2-pentanone and Methylene Chloride) may be present above 5 times the EQL (Estimated Quantitaion Limit).

2. Target compounds other than common solvents must not be greater than the reporting limit.

3. Non-target compound peaks with areas greater than 10% of the area of the nearest internal standard, other than surrogates, must not be present in the chromatogram if tentatively identified compounds (TICS) are requested.

If contaminants are found in the blank, an attempt should be made to identify and minimize/eliminate the source.

D. Lab Control Sample

A lab control sample (LCS) and lab control sample duplicate (LCSD) are performed within every 12 hr. analytical clock to check for instrument accuracy and precision. Prepare a 50 ug/L LCS/LCSD by adding 25 uL of the second source spike calibration mix at 100 ug/mL into a 50 mL voulmetric. Invert the volumetric three times slowly and pour contents into a 40 mL non-preserved VOA vial and place on the autosampler. The autosampler will then add 1 uL of ISSTD mix @ 250 ug/mL into a 5 mL aliquot. The spike compounds** and the specified recovery limits are as follows:

Compound	Aqueous
1,1-Dichloroehtene	61-145
Trichloroethene	71-120
Benzene	76-127
Toluene	76-125
Chlorobenzene	75-130

* All limits are taken from CLP OLM3.0 criteria.

** An LCS for all compounds may be required based on project requirements and scope. If a LCS with all target analytes is requested, a LCSD may not be analyzed. The calibration mix used for the full spike LCS is the same used for the MS/MSD.

E. Blank Spike/Blank Spike Duplicate

A blank spike(BS)/blank spike duplicate(BSD) will be used for QC control in methanol extracted soils if Matrix Spike/Matrix Spike Duplicates are not analyzed because of insufficient sample amounts. A BS/BSD is made by weighing 4g of Ottowa sand and 4 mLs of P & T methanol into a 20 mL scinillation vial. 4 uL of the surrogate mix @ 2500 ug/mL and 125 uL of a second source spike mix @ 100 ug/mL is spiked into the scinillation vial giving the surrogates and spikes a final concentration of 50 ug/L. A 1 mL methanol aliquot of the sample is placed into a 50 mL volumetric and brought to volume with OFW. The volumetric is inverted three times slowly, poured into a non-preserved 40 mL VOA vial and placed on the instrument for analysis. The autosampler then adds 1 uL of the ISTD mix @ 250 ug/mL into a 5 mL sample aliquot for a final ISTD concentration of 50 ug/L.

Due to poor purge efficiencies of some compounds found in the spike mixes, a default percent recovery criteria of 70 to 130 has been established.

IV. Sample Analysis

A. Identify Matrix

The two matrices are water and methanol extracted soils. A methanol extract is required if a 5.0 g sample size of soil yielded results above the calibration range.

B. Sample Preparation

Samples must be warmed to ambient temperature before analysis begins.

1. Water:

A). A 40 mL VOA vial of the specified sample is placed onto the autosampler. A 5 mL aliquot of the sample is transferred into a sparge tube and purged.

B). Primary Dilution (1:10 or less) - 500 uL to 2.5 mL of sample is added into OFW on the autosampler for a final volume of 5mL.

C). Secondary Dilution (1:20 to 1:10,000) - 5.0 µL to 2.5 mL of sample is added into a 50 mL volumetric brought to volume with OFW. Invert slowly three times. Pour the contents into a non-preserved 40 mL VOA vial and place on the instrument for analysis.

To each 5 mL sample aliquot, the autosampler adds 1 µL of the ISSTD mix at 250 ug/L, resulting in a final concentration of 50 µg/L.

2. Soil:

A). Medium Level Extracts

A 25g En Core sampler is extruded into 20 mLs of Purge and Trap Methanol in a 60 mL soil jar. The tar weight of the jar and the approximate weight of 20 mLs of MEOH (15.8g) is added and recorded. The jar containing the 25g plug then placed on the balance and the tare weight recorded. The final sample weight is found by subtracting the weight of the jar/MEOH from the weight of the jar/MEOH/sample. Methanol is then added to bring the final ratio of soil to MEOH to 1:1. The amount of surrogate @ 2500 ug/mL added is equal to the final weight of the sample/MEOH.

A 5g En Core may also be used in place of a 25g En Core sampler. It will follow the same prcodure as mentioned above except the sample will be extruded into a 20 ml scinillation vial containing 3 mLs of P&T MEOH. The most important aspect is to maintain a 1:1 ratio of soil to methanol.

Samples received pre-weighed in the field should also have a 5g or 25g sample aliquot already preserved in MEOH. The same procedure for medium level preperation, as listed above, is followed.

QC samples are prepped per 20 samples. A MEOH blank is made by weighing 5g of Ottowa sand into a 20 mL scinillation vial and adding 5 mLs of P & T methanol. In order to obtain a final concentration of 50 ug/L for surrogates, 5 uL of surrogate mix @ 2500 ug/mL is spiked into the scinillation vial. The same process is used for Blank Spikes/Blank Spike Duplicates* except 4g of Ottowa sand and 4 mLs of P & T methanol are placed into a 20 mL scinillation vial. 4 uL of the surrogate mix @ 2500 ug/mL and 125 uL of a second source spike mix @ 100 ug/mL is spiked into the scinillation vial giving the surrogates and spikes a final concentration of 50 ug/L.

A 1 mL methanol aliquot of the sample is placed into a 50 mL volumetric and brought to volume with OFW. The volumetric is inverted three times slowly, poured into a non-preserved 40 mL VOA vial and placed on the instrument for analysis. The autosampler then adds 1 uL of the ISTD mix @ 250 ug/mL into a 5 mL sample aliquot for a final ISTD concentration of 50 ug/L.

Record all data pertinent to medium level soil extract preparation in the designated logbook.

* The BS/BSD is used when MS/MSD samples are not available due to insufficient sample amounts.

C. Analysis

Analyze samples by purge-and-trap GC/MS methodology. See En Chem SOP for proper instrument operating conditions. Waters and medium level soils are purged at ambient temperature.

Identify and quantitate the target compounds using relative retention times (RRT) and average response factors from the 5-point calibration curve. For establishing correspondence of the RRT, the sample component RRT must compare within +/-0.06 RRT units of the RRT of the continuing calibration standard component. Compare computer-matched compounds with reference spectra to accept or reject each identification. All ions present in the reference spectrum that are at least 10% of the base peak must be present in the sample background-subtracted spectrum. Also, the relative intensities of these ions must agree within +/-20% between the standard and sample spectra. While this is a good guideline, acceptance or rejection will depend upon the judgement of the analyst.

It may be necessary to adjust the volume of sample used for analysis so that the concentration of the analytes of interest are within the calibration range. A maximum of 5.0 mL of an aqueous sample will be analyzed. While every attempt is made to provide the best detection limits, the most concentrated analysis of a sample is often limited by interfering peaks of non-target compounds. Detection limits will be raised to account for the dilution.

D. Verification of sample preservation

Following sample analyses, the pH of all waters shall be verified by wide range pH paper. The results will be documented in the logbook. If the pH is found to be 3 or greater and the sample was not analyzed within 7 days of collection, a narravtive will written and submitted with the sample results.

Quality Control:

The surrogate recoveries and internal standard areas must be checked to determine that they are within the established control limits. If the requirements are not met, the sample is reanalyzed. The internal standard areas for the method blank and samples analyzed must be within -50% to +100% of the calibration check standard's internal standard areas. If an internal standard or surrogate is outside of control limits but no compounds of interest are being quantified, no re-analysis is necessary. If the second analysis is within control limits, the data from that analysis is reported. If the failure is repeated, data from the "better" of the two analyses is reported and the anomaly is discussed in a sample narrative.

A sample matrix spike (MS) and matrix spike duplicate (MSD) must be performed every 14 days or 20 samples per instrument of the same matrix type by the same method and target compound list. A matrix type for an MS/MSD is defined as an unheated water which includes medium level soil methanol extracts. The spiking solution must contain all constituents of concern and be from a secondary source other than the reference standards used in the calibration. The percent recoveries for each spiked compound must be checked to determine that they are within the established control limits. If these are not met, then the sample MS and/or MSD is reanalyzed.

E. Annual Quality Control

1. Method Detection Limits (MDLs):

MDLs are determined as specified in 40 CFR, Part 136, Appendix B and must be performed once a year per instrument for all target compounds of interest. Each available instrument will perform one unheated set of MDL's using Purge and Trap methodology. At least seven replicates will be analyzed per set.

2. Surrogate Recovery Limits:

One set each of surrogate recovery limits for unheated waters and methanol extracted soils must be established for all instruments performing GC/MS analysis. These limits are to be updated annually using data from all GC/MS instruments.

3. Matrix Spike and Duplicate (MS/MSD) Recovery Limits:

One set each of MS/MSD recovery limits for unheated waters and methanol extracted soils must be established for all instruments performing GC/MS analysis. These limits are to be updated annually using data from all GC/MS instruments.

APPENDIX A

FORMULA SHEET

#1 RESPONSE FACTOR:

$$RF = \text{RESPONSE FACTOR} = \frac{(Ax)}{(Ais)} \times \frac{(Cis)}{(Cx)}$$

where:

Ax = Area of analyte's characteristic ion of quantitation

Ais = Area of corresponding internal standard's ion of quantitation

Cis = Concentration of corresponding internal standard

Cx = Concentration of analyte to be measured

#2 Calculation for Raw amount using Average RF and Internal Standards

$$\text{Raw amount} = \frac{(Ax)}{(Ais)} \times \frac{(Cis)}{\text{Avrg RF}}$$

where:

Ax = Area of analyte's characteristic ion of quantitation

Ais = Area of corresponding internal standard's ion of quantitation

Cis = Concentration of corresponding internal standard

Avrg RF = Average Response factor of compound from initial calibration

APPENDIX B

TARGET COMPOUND LIST, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQL's)

	LOW LEVEL EQL*	MEDIUM LEVEL EQL
Dichlorodifluoromethane	2	50
Chloromethane	2	50
Bromomethane	2	50
Vinyl chloride	2	50
Chloroethane	2	50
Trichlorofluoromethane	2	50
Dichlorofluoromethane	2	50
Allyl Chloride	5	250
Acrolein	10	500
Acetone	5	150
Diethyl Ether	1	50
1,1-Dichloroethene	1	50
Iodomethane	5	250
Acrylonitrile	10	500
Methylene chloride	1	50
Carbon disulfide	1	50
Trans-1,2-Dichloroethene	1	50
Methyl Tert Butyl Ether	1	50
1,1-Dichloroethane	1	50
Vinyl Acetate	10	500
2-Butanone	5	150
Diisopropyl Ether	1	50
Cis-1,2-Dichloroethene	1	50
Bromochloromethane	1	50
Chloroform	1	50
2,2-Dichloropropane	1	50
Tetrahydrofuran	10	500
1,2-Dichloroethane	1	50
1,1,1-Trichloroethane	1	50
1,1-Dichloropropene	1	50
Carbon tetrachloride	1	50
Benzene	1	50
Diethoxymethane	10	500
Dibromofluoromethane (SURR)		
Trichloroethene	1	50
1,2-Dichloropropane	1	50
2,3-Dichloropropane	1	50
Dibromomethane	1	50
Bromodichloromethane	1	50
2-chloroethyl vinyl ether	5	250

cis-1,3-Dichloropropene	1	50
Toluene	1	50
trans-1,3-Dichloropropene	1	50
4-Methyl-2-pentanone	5	150
1,1,2-Trichloroethane	1	50
Tetrachloroethene	1	50
1,3-Dichloropropane	1	50
Dibromochloromethane	1	50
2-Hexanone	5	150
1,2-Dibromoethane	1	25
Toluene-d8 (SURR)		
Chlorobenzene	1	50
1,1,1,2-Tetrachloroethane	1	50
Ethylbenzene	1	50
m-, p-, Xylene	2	100
o-Xylene	1	50
Styrene	1	50
Bromoform	1	50
Isopropylbenzene	1	50
Trans-1,4-Dichloro-2-Butene	5	250
Cis-1,4-Dichloro-2-Butene	5	250
Bromobenzene	1	50
4-Bromofluorobenzene (SURR)		
1,1,2,2-Tetrachloroethane	1	50
1,2,3-Trichloropropane	1	50
n-Propylbenzene	1	50
2-Chlorotoluene	1	50
4-Chlorotoluene	1	50
1,3,5-Trimethylbenzene	1	50
Tert-Butylbenzene	1	50
1,2,4-Trimethylbenzene	1	50
Sec-Butlybenzene	1	50
1,3-Dichlorobenzene	1	50
1,4-Dichlorobenzene	1	50
1,2,3-Trimethylbenzene	1	50
P-Isopropyltoluene	1	50
1,2-Dichlorobenzene	1	50
N-Butylbenzene	1	50
1,2-Dibromo-3-chloropropane	5	250
1,2,4-Trichlorobenzene	1	50
Naphthalene	1	50
Hexachlorobutadiene	1	50
1,2,3-Trichlorobenzene	1	50

*Lower quantitation limits may be obtained depending on project requirements.

APPENDIX C

TABLE 1
SW-846
GC/MS PERFORMANCE STANDARD

Bromofluorobenzene (BFB)

m/z	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

TABLE 2
CLP OLM3.1
GC/MS PERFORMANCE STANDARD

Bromofluorobenzene (BFB)

m/z	Ion Abundance Criteria
50	8-40% of mass 95
75	30-66% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	50-120% of mass 95
175	4-9% of mass 174
176	93-101% of mass 174
177	5-9% of mass 176

TABLE 3

Calibration Check Compounds (CCC)

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

* The % RSD for the initial calibration must be less than 30%.
* The % RSD for the continuing calibration must be less than 20%.

System Performance Check Compounds (SPCC)

	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
Chlorobenzene	0.30

En Chem, Inc.

Quality Assurance Document

SET No: 1

Standard Operating Procedure

TITLE: Extraction of Water Samples for Base/Neutral/Acids

DEPARTMENT: Semivolatile Organics Extractions

REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 Method 3510C (Dec. 1996)

Code of Federal Regulations
USEPA 40CFR (1988), Pt.136, App.A, Method 625

PROCEDURE SUMMARY:

A measured volume of water sample, approximately one liter, is serially extracted with methylene chloride at a pH less than 2, and again at a pH greater than 11, using a separatory funnel. The methylene chloride extracts are dried, combined and concentrated to a volume of 1 mL.

REVIEWED BY: Daniel M Rude — Date: 2-3-98
Daniel M. Rude
Group Leader
Organic Laboratory

Gregory J. Graf — Date: 2-3-98
Gregory J. Graf
Quality Assurance Officer

APPROVED BY: Eric L Thomas — Date: 2/6/98
Eric L. Thomas
Laboratory Manager

QUALITY CONTROL:

- The sample holding time is 7 days from the date of sampling.

- One method blank is extracted per 20 samples OR per extraction batch whichever is more frequent. Reagent method blanks are prepared from laboratory de-ionized water.

- Surrogate standards should be added to all samples, laboratory control spikes, matrix spikes, and method blanks. Surrogates are used to monitor unusual matrix effects, sample processing problems, etc.

- A matrix spike and matrix spike duplicate will be performed for every 20 samples. If insufficient sample or unsuitable matrix does not allow this, there will be two laboratory control spikes performed instead for every 20 samples. The time frame for the 20 samples cannot extend beyond 14 days. Matrix spike compounds are used to indicate the presence or absence of unusual matrix effects.

- A laboratory control spike should be performed with the matrix spike and the matrix spike duplicate for every 20 samples. The time frame for the 20 samples cannot extend beyond 14 days. Control spikes are prepared from laboratory de-ionized water.

INTERFERENCES:

Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

MATERIALS AND APPARATUS:

Separatory funnel:	2000 mL with Teflon stopcock.
Concentrator tube:	Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent).

Evaporation flask:	Kuderna-Danish, 500 mL (Reliance R-3705-030 or equivalent). Attach to concentrator tube with clips.
Snyder columns:	Kuderna-Danish, three-ball macro (Kontes K-503000-0121 or equivalent).
	Kuderna-Danish, two-ball or three ball micro (Kontes K-569001-0219 or equivalent).
Vials:	Amber glass, 12 mL capacity with teflon-lined screw cap. 2.0 mL clear injection vials with crimp caps.
Funnel:	150-gram capacity.
Glass wool:	Rinsed with methylene chloride.
Boiling chips:	Solvent rinsed and dried, approximately 10/40 mesh (silicon carbide or equivalent).
Sodium Sulfate:	Preheated at 400° C for 4 hours in a crucible.
Water bath:	Heated, with concentric ring cover, capable of temperature control (±5° C). The bath should be used in a hood.
Tube heater:	Kontes 720000-0000.
Pipets:	Disposable, 2 mL short-stem.
Syringes:	500 µL and 1000 µL Gastight syringes (Hamilton 1000 series or equivalent).
pH paper:	Wide range pH paper.
Graduated Cylinder:	1000 mL.

REAGENTS:

Surrogate Spiking Solution: See appendix A.

Matrix Spiking Solution: See appendix B.

Sodium hydroxide (NaOH): 10 Normal; dissolve 80 g of NaOH pellets in reagent water and dilute to 200 mL.

Sulfuric Acid (H_2SO_4): (1:1); slowly add 50 mL concentrated H_2SO_4 (specific gravity 1.84) to 50 mL reagent water.

Methylene chloride pesticide grade or equivalent.

EXTRACTION PROCEDURE OUTLINE:

1 Allow the sample to warm to room temperature, mark the sample volume on the sample container, then invert sample several times.

2 Set up separatory funnel, K-D apparatus, and funnel with glass wool and sodium sulfate. Silicon carbide boiling chips will be added just prior to concentration.

2a Label the glassware with the sample number.

3 Transfer sample volume to separatory funnel.

4 Rinse sample container with 60 ml of methylene chloride and transfer solvent rinse to separatory funnel. **Record methylene chloride lot # on the extraction form.**

5 Adjust sample pH to <2 with 1:1 sulfuric acid. Check with wide range pH paper.

6 Add the required amount (1000 µL) of surrogate solution to each separatory funnel with a 1000 µL syringe. **Record the amount of surrogate solution used and the reference number on the surrogate solution vial on the extraction form.**

7 Add the required amount (1000 µL) of matrix spike solution only to those appropriate samples with a 1000 µL syringe. **Record the amount of spiking solution used and the reference number on the matrix spike solution vial on the extraction form.**

8 Shake separatory funnel for 1 to 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase. If an emulsion interface occurs between the phases, the technician must employ mechanical techniques to complete phase separation.

NOTE: Methylene chloride creates excessive pressure very rapidly: therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Venting of the separatory funnel should be into a hood to avoid needless exposure of the technician to solvent vapors.

9 Drain the extract through the funnel containing glass wool and sodium sulfate. Collect the extract in the K-D apparatus. Rinse the sodium sulfate with methylene chloride.

10 Add 60 mL methylene chloride to the separatory funnel and repeat steps 8 through 9. This process is repeated one more time.

11 Adjust sample pH to >11 with 10 N sodium hydroxide. Check with wide range pH paper.

12 Add 60 mL methylene chloride to separatory funnel, shake the funnel for 1 to 2 minutes with periodic venting to release excess pressure, allow the layers to separate, and drain extract through funnel containing glass wool and sodium sulfate into the K-D apparatus containing the acid fraction. Rinse the sodium sulfate with methylene chloride.

13 Repeat step 12 two times. The total extract volume in the K-D apparatus should be approximately 400 mL.

14 Attach a Snyder three-ball column to K-D apparatus and set aside for future concentration.

15 Determine initial sample volume. Fill the original sample container from step 1 to the mark with water. Transfer contents to a 1000 mL graduated cylinder. Record the initial sample volume on the extraction form to the nearest 5 mL.

SAMPLE EXTRACT CONCENTRATION :

1 Place the K-D apparatus on a hot water bath (70° to 75° C) so that the concentrator tube is totally immersed in the hot water and the entire lower rounded surface of the flask is bathed with water. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 3 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

2 Rinse the Snyder column and the evaporating flask with a small amount of methylene chloride. Remove Snyder column and evaporating flask.

3 Final concentration of the sample extract can be performed by either the Nitrogen Blowdown method or the Micro-snyder column method as follows:

NITROGEN BLOWDOWN CONCENTRATION METHOD:

1 Concentrate each sample to approximately 0.8 mL on the Kontes tube heater (set at 40°C) with dry nitrogen. Rinse the internal wall of the concentrator tube several times with methylene chloride during the nitrogen blowdown. <u>DO NOT ALLOW THE EXTRACT TO BECOME DRY, OR NEARLY DRY AS THIS WILL RESULT IN LOSS OF ANALYTES.</u>

2 Adjust to a final volume of 1.0 mL using methylene chloride by rinsing down the walls of the concentrator tube. Transfer 1.0 mL of the extract to a prelabelled injection vial and cap with an injection cap.

3 Label the vial with project name, extraction date, sample number, final volume.

4 Log sample into the analysts' extract storage refrigerator and complete all paperwork.

MICRO-SNYDER COLUMN CONCENTRATION METHOD:

1 Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube. Place the apparatus in the hot water bath. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the liquid reaches an apparent volume of 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the Snyder column and rinse its lower joint with approximately 0.2 mL of appropriate solvent into the concentrator tube. DO NOT ALLOW THE EXTRACT TO BECOME DRY, OR NEARLY DRY AS THIS WILL RESULT IN LOSS OF ANALYTES.

2 Adjust to a final volume of 1.0 mL using methylene chloride by rinsing down the walls of the concentrator tube. Transfer 1.0 mL of the extract to a prelabelled injection vial and cap with an injection cap.

3 Label the vial with project name, extraction date, sample number, final volume.

4 Log sample into the analysts' extract storage refrigerator and complete all paperwork.

Appendix A
SPIKING STANDARDS FOR BASE/NEUTRAL/ACIDS

SURROGATE SPIKING SOLUTION

The mixture contains the following components at:

150 µg/mL	100 µg/mL
2-Fluorophenol	Terphenyl-d14
2,4,6-Tribromophenol	2-Flourobiphenyl
Phenol-d5	1,2-Dichlorobenzene-d4
2-Chlorophenol-d4	Nitrobenzene-d5

Appendix B
SPIKING STANDARDS FOR BASE/NEUTRAL/ACIDS

MATRIX SPIKING SOLUTION

The mixture contains the following components at 100 µg/mL:

Phenol
2-Chlorophenol
Benzyl alcohol
2-Methylphenol
4-Methylphenol
2,4-Dimethylphenol
Benzoic acid
2,4-Dichlorophenol
4-Chloroaniline
4-Chloro-3-methylphenol
2-Nitrophenol
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
2-Nitroaniline
3-Nitroaniline
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran
4-Nitroaniline
1,2-Diphenylhydrazine
Pentachlorophenol
Benzidine
3,3'-Dichlorobenzidine
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene
2-Methylnaphthalene
1-Methylnaphthalene
Butylbenzylphthalate
Di-n-butylphthalate
bis(2-Ethylhexyl)phthalate
Di-n-octylphthalate
Phenanthrene
Anthracene
Pyridine
N-nitrosodimethylamine
bis(2-Chloroethyl)ether
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
2,2'-oxybis(1-Chloropropane)
N-nitroso-di-n-propylamine
Hexachlorethane
Nitrobenzene
Isophorone
Carbazole
bis(2-Chloroethoxy)methane
1,2,4-Trichlorobenzene
Napthalene
Hexachlorobutadiene
2-Chloronaphthalene
Dimethylphthalate
Acenaphthylene
2,6-Dinitrotoluene
Acenaphthene
2,4-Dinitrotoluene
Diethylphthalate
4-Chlorophenyl-phenylether
Fluorene
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
4-Bromophenyl-phenylether
Hexachlorobenzene
Fluoranthene
Benzo(a)anthracene
Chrysene
Pyrene
Benzo(b)fluoranthene
Benzo(k)fluoranthene

En Chem, Inc.

Quality Assurance Document

Standard Operating Procedure
Analytical Method

TITLE: Analysis of Base/Neutral and Acid (BNA) Compounds by GC/MS

DEPARTMENT: Semivolatile Organics

APPLICATION: This method is used to determine the concentration of various BNA compounds in water, solid waste and biological tissue samples Appendix A contains the compounds that may be determined by this method and the detection limits for each compound in reagent water.

REFERENCES. Test Methods for Evaluating Solid Wastes
SW846 method 8000B (Dec 1996)
SW846 method 8270C (Dec 1996)

Code of Federal Regulations
USEPA Method 625 40CFR Pt 136, App A, Ch 1 (7-1-88 Ed)

PROCEDURE SUMMARY:

This method provides the gas chromatographic conditions for the separation of the compounds in the extract for the quantitative analysis by mass spectrometry. A volume of a sample extract is injected into a gas chromatograph (GC) and compounds in the GC effluent are analyzed by mass spectrometry (MS).

REVIEWED BY: Daniel M Rude | Date: 10-13-2000
Daniel M. Rude
Semivolatile Group Leader

Gregory J Graf | Date: 10-13-2000
Gregory J. Graf
Quality Assurance Officer

APPROVED BY: Glen A. Coder | Date: 10-13-2000
Glen A. Coder
Laboratory Manager

SAMPLE EXTRACT HANDLING AND STORAGE

Store all extracts at 4° ± 2° C in the dark in Teflon-sealed containers until analysis is complete. Sample extracts <u>must</u> be analyzed within 40 days from time of extraction

INTERFERENCES·

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided

Matrix interference's may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference's will vary considerably from source to source The GPC(Gel Permeation Chromotography) cleanup procedure is available for cleaning up the sample extract. The need for GPC cleanup is initially determined by the extraction personnel. The analyst can request that the sample extracts have GPC cleanup prior to the addition of Internal Standards. This is usually determined by visual inspection of the sample extract or by historical data. Tissue samples should routinely be cleaned by GPC.

APPARATUS AND MATERIALS·

GC/MS: Hewlett Packard (HP) GC5890 series / MSD5970, MSD5972 or equivalent, capable of scanning 35-500 amu at 1 sec/scan.

GC Autosampler: HP7673 or equivalent.

Data Processor: HP ChemStation (acquiring) / HP ChemServer-Target 3 (analysis) or equivalent.

Printer: HP Laserjet 4 or equivalent

Syringes: 10-1000 µL Gastight syringes (Hamilton series 1000 or equivalent).

Autosampler Vials: 2 mL with crimp top caps.

GC Column: Rtx-5MS capillary column, 30 m x 0.32 mm I.D. x 0.5 um df with guard column or XTI-5 capillary column, 30 m x 0 25 mm I.D. x 0.25 um df with guard column(Restek or equivalent).

GC Column Conditions: Carrier gas - Helium
Flow rate - 1.2 mL/min
Linear velocity - 43.1 cm/sec
Detector temp. -290° C
Injector temp - 280° C
Splitless Injection Flow rate - 50-60 mL/min.
Auxillary E pressure control - 50 psi

Inlet B Pressure Program:

Initial Pressure	- 0 2 psi
Initial Time	- 0 10 min
Level 1 Rate	- 99 psi/min.
Final 1 Pressure	- 10 psi
Final 1 Time	- 0.40 min.
Level 2 Rate	- 99 psi/min.
Final 2 Pressure	- 0.2 psi
Final 2 Time	- 1.2 min.
Level 3 Rate	- 0.3 psi/min.
Final 3 Pressure	- 10 psi
Final 3 Time	- 0.0 min.

GC Temperature Program

Initial temp -	40° C
Initial time -	2 min.
Rate 1 -	10° C/min.
Final 1 temp. -	300° C
Final 1 time -	0.0 min.
Rate 2 -	16 C/min
Final 2 temp. -	320 C
Final 2 time -	7 min.

REAGENTS:

Solvents: Methylene chloride and acetone pesticide grade.

Stock Standards Solutions: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 12 months from the date of preparation.

Calibration Standards: Standard Mixtures containing all compounds including surrogate compounds at 6 concentration levels are prepared from the stock solutions. Each calibration solution is spiked with 40 ng of internal standard solution. One of the concentration levels should be at a concentration near, but above, the method detection limit. Shelf-life of the calibration solutions is 6 months from the date of preparation.

Internal Standards. A commercially prepared standard mix at a concentration of 4000 ug/mL is used. This solution is certified by the manufacturer (Restek). Shelf-life of standard solution is 6 months from the date of preparation. See Appendix B.

Surrogate Standards	Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation. See appendix B
Matrix Spike/Laboratory Control Sample Standards:	A commercially prepared stock standard solution is used at a concentration of 100 ug/mL certified by the manufacturer See appendix C for the list of compounds in the matrix spike mix. See appendix G for the list of compounds in the Laboratory Control Sample spike mix
GC/MS Tuning Standard:	Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation. See appendix B.

GC/MS INITIAL CALIBRATION:

1. GC/MS tuning standard. Inject 1 uL of 50 ng of Decafluorotriphenylphosphine (DFTPP) The average of three scans (the apex and the scan before and after the apex) may be used. Background subtraction is required and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. Compare the mass listing to the tuning criteria in appendix D. The tuning criteria <u>must</u> be met in order to continue calibration or sample analysis.

All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

The DFTPP tuning standard should include 50 ng each of the following additional compounds, pentachlorophenol, benzidine, and DDT in order to assess GC column performance and injection port inertness. The responses for benzidine and pentachlorophenol should be normal with no peak tailing. The degradation of DDT to DDE and DDD should not exceed 20%. If degradation of DDT is excessive and/or the chromatography for benzidine or pentachlorophenol is poor the injection port may require cleaning and replacement of the glass liner, liner insert, and the gold seal. Also 6-12 inches of the column should be cut off. This preventative must be performed prior to sample analysis. If the response for pentachlorophenol continues to be very poor or absent then the column may need to be replaced.

Total peak area of

$$\%\ \text{DDT Breakdown} = \frac{(\text{DDD} + \text{DDE})}{(\text{DDD} + \text{DDE} + \text{DDT})} \times 100\%$$

2. Inject 1 µL of each of the calibration standard solutions, SSTD010, SSTD020, SSTD050, SSTD080, SSTD120, and SSTD160. Determine the response factors (RF), the average RF and percent relative standard deviation (%RSD) for each compound.

   a. $RF = (A_x C_{is})/(A_{is} C_x)$

      where A_x = Area of the characteristic ion for the compound being measured

      A_{is} = Area of the characteristic ion for the specific internal standard

      C_{is} = Concentration of the specific internal standard

      C_x = Concentration of the compound being measured

   b. %RSD = 100[SD/RF]

      The %RSD should be less than or equal to 15% for each compound The %RSD must not exceed 30% for the Calibration Check Compounds (CCC) (See Appendix D).

      Linearity - If the %RSD is less than or equal to 15%, then the average RF is used for calculating the concentration of the compound being measured If the %RSD is greater than 15% then the analyst should select the regression order which introduces the least error into the quantitation.

   c. The System Performance Check Compounds (SPCC) must have a minimum RF of 0.050 (See Appendix D)

      If these criteria are not met, corrective action is required such as cleaning or replacing the injection port liner and/or capillary column.

      If the CCCs are not included in the list of analytes for a project, then all required analytes must meet the 30% RSD criterion.

Analysis of Initial Calibration Verification Standard

May be required based on Project Requirements.

1. In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed within the same time clock as the calibration curve The ICV standard must be from a second source stock and meet the same criteria as the Continuing Calibration Verification (CCV) standard before the initial calibration may be considered valid.

GC/MS Daily Calibration:

1 GC/MS Tuning Standard (DFTPP) Inject 1 µL of 50 ng DFTPP and compare the mass listing to the acceptance criteria in Appendix D. The tuning standard <u>must</u> precede each 12 hour analysis sequence.

2 A midpoint calibration standard (50 ppm) must precede sample analysis. The calibration check response factors are compared to average response factors from the initial calibration.

a. The SPCCs <u>must</u> meet the minimum RF criteria of 0.050

b. The CCCs <u>must</u> meet the percent difference (%D) criteria

$$\%D = \frac{RF_{ave} - RF}{RF_{ave}} X100$$

If the percent difference for any compound is greater than 20%, this is considered a warning limit If the percent difference for each of the CCCs is less than or equal to 20%, then the initial calibration is assumed valid and analysis of samples may proceed If the 20% criterion is not met, corrective action <u>must</u> be taken in either instrument maintenance or re-calibration. If the CCCs are not analytes required, then all required analytes <u>must</u> meet the 20% drift criterion

If the retention time for any internal standard in the continuing calibration check standard changes by more than 30 seconds from the last calibration check (12 hours), then the chromatographic system must be inspected for any malfunction and corrective action must be made.

SAMPLE ANALYSIS·

1. All samples, method blanks, laboratory control samples and matrix spikes <u>must</u> be analyzed within 12 hours of a valid DFTPP tuning standard

2. All samples, method blanks, laboratory control samples, and matrix spike extracts are spiked with 40 ng of internal standard solution mix just prior to analysis

The internal standard areas of the samples, method blanks, laboratory control samples, and matrix spikes must fall within a factor of two (-50% to +100%) range from the preceding midpoint calibration check standard. In addition, the relative retention times of the internal standards for each sample analysis must fall within a ± 30 second window defined by the midpoint calibration check standard.

3. Surrogate recoveries are calculated using the following equation.

Surrogate % Recovery = $(C_{ex} / C_s) \times 100$

where: C_{ex} = Concentration of analyte in the extract (mg/L).
C_s = Calculated concentration of analyte spiked into extract based on amount spiked (mg/L).

Compare the surrogate recoveries according to the specific matrix to the recovery limits in appendix E. These limits are updated annually.

4. Qualitative sample analysis:

   a. The relative retention time (RRT) for the sample component must compare within ± 0.06 RRT units of the standard component.

   b. The mass spectrum for a sample component should compare to the spectrum of the standard component. Note: These criteria do not overrule the judgment of the analyst.

      1. All ions present in the standard mass spectrum greater than 10% should be present in the sample spectrum
      2. The relative intensities of those ions must agree within ± 30% of those ions in the reference spectrum

5. Quantitative sample analysis:

When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. See Appendix A for primary ion (1°) of each compound

Water:
$$\text{concentration } (\mu g/L) = \frac{(C_{ex})(V_F)(DF)}{(V_o)}$$

Soil/Tissue.
$$\text{concentration } (\mu g/kg) = \frac{(C_{ex})(V_F)(DF)}{(W_s)(D)}$$

where: C_{ex} = Concentration of analyte in the extract (ug/mL)
DF = Dilution factor (if applicable)
V_o = Initial sample volume (L)
V_F = Final extract volume (mL)
W_s = Initial sample weight extracted (kg)
D = % solids

QUALITY CONTROL

1. The method blank must meet the surrogate limits (see appendix E). If the blank fails this criteria, all of the associated samples, matrix spikes and laboratory control spikes must be re-extracted.

2. If the blank contains any analyte of interest above the reporting limit (except the phthalate esters, see appendix A), all of the associated samples, matrix spikes, and laboratory control spikes must be re-extracted. **NOTE:** A method blank for semivolatile analysis must contain less than five times (5x) the required quantitation limit (EQL) of the phthalate esters listed in Appendix A.

3. The sample surrogate recovery must meet the surrogate limits (see appendix E). Allowance is made for one acid extractable and/or one base/neutral extractable surrogate to be out. If any sample fails this criteria, the sample must be re-extracted unless it is demonstrated to be a matrix effect

4 Every batch of samples must contain a Laboratory Control Sample (LCS). The LCS is used to verify method performance in the event of poor recoveries in the Matrix Spike or Matrix Spike Duplicate.

   The control limits for the LCS should fall within the prescribed limits (see Appendix G). A small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger reextraction and analysis of the entire batch). See Appendix I for amount of sporadic failures allowed.

5. Sample matrix spike component recoveries should fall within the prescribed limits (see Appendix F).

   If any sample matrix spike component fails the recovery criterion, the following should be implemented.

   a. If the laboratory control sample analyzed in the sequence satisfies the recovery criterion as specified in appendix G, no re-extraction of the sample matrix spike is required.

   b If the matrix spike recovery failure is duplicated in the matrix spike duplicate, no re-extraction is required

   c. A small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger re-extraction and analysis of the entire batch). See Appendix I for allowances for sporadic failures.

6 Leachate samples (TCLP) have different quality control limits for surrogate percent recoveries, LCS percent recoveries and MS percent recoveries See Appendix E for the surrogate criteria and Appendix H for the LCS and MS criteria

Appendix A
DETECTION LIMITS
for BNAs

Compound	ENCHEM[a] Method Detection Limit (µg/L)	ENCHEM[b] Reporting Limit Water (µg/L)	ENCHEM[b] Reporting Limit Solid (µg/kg)	1° Ion
Phenol	0.18	10	330	94
bis(2-Chloroethyl)ether	0.29	10	330	63
2-Chlorophenol	0.32	10	330	128
1,3-Dichlorobenzene	0.31	10	330	146
1,4-Dichlorobenzene	0.41	10	330	146
1,2-Dichlorobenzene	0.38	10	330	146
2-Methylphenol	0.21	10	330	108
2,2'-oxybis(1-Chloropropane)	0.27	10	330	45
4-Methylphenol	0 20	10	330	108
N-Nitrosodi-n-propylamine	0.22	10	330	70
Hexachloroethane	0.55	10	330	117
Nitrobenzene	0 34	10	330	77
Isophorone	0.22	10	330	82
2-Nitrophenol	0.32	10	330	139
2,4-Dimethylphenol	0.30	10	330	107
bis(2-Chloroethoxy)methane	0.24	10	330	93
2,4-Dichlorophenol	0.25	10	330	162
1,2,4-Trichlorobenzene	0.35	10	330	180
Naphthalene	0.27	10	330	128
4-Chloroaniline	0 38	10	330	127
Hexachlorobutadiene	0.56	10	330	225
4-Chloro-3-methylphenol	0.32	10	330	107
2-Methylnaphthalene	0.26	10	330	142
Hexachlorocyclopentadiene	1.23	10	330	237
2,4,6-Trichlorophenol	0.31	10	330	196
2,4,5-Trichlorophenol	0.41	25	830	196
2-Chloronaphthalene	0.26	10	330	162
2-Nitroaniline	0 23	25	830	65
Dimethylphthalate	0.15	10	330	163
Acenaphthylene	0.22	10	330	152
2,6-Dinitrotoluene	0 20	10	330	165
3-Nitroaniline	0.23	25	830	138
Acenaphthene	0.24	10	330	154
2,4-Dinitrophenol	6.44	25	830	184
4-Nitrophenol	0.92	25	830	109
Dibenzofuran	0.20	10	330	168
2,4-Dinitrotoluene	0.29	10	330	165
Diethylphthalate	0.31	10	330	149

Appendix A (Continued)
DETECTION LIMITS
for BNAs

Compound	ENCHEM[a] Method Detection Limit (µg/L)	ENCHEM[b] Reporting Limit Water (µg/L)	Solid (µg/kg)	1° Ion
4-Chlorophenyl phenyl ether	0.22	10	330	204
Fluorene	0 24	10	330	166
4-Nitroaniline	0.31	25	830	138
4,6-Dinitro-2-methylphenol	1.75	25	830	198
N-Nitrosodiphenylamine	0 23	10	330	169
4-Bromophenyl phenyl ether	0 23	10	330	248
Hexachlorobenzene	0 31	10	330	284
Pentachlorophenol	3.13	25	830	266
Phenanthrene	0 25	10	330	178
Anthracene	0.17	10	330	178
Carbazole	0 25	10	330	167
Di-n-butylphthalate	0.35	10	330	149
Fluoranthene	0.22	10	330	202
Pyrene	0.23	10	330	202
Butylbenzylphthalate	0.32	10	330	149
3,3'-Dichlorobenzidine	0.44	10	330	252
Benzo(a)anthracene	0.20	10	330	228
Chrysene	0.19	10	330	228
bis(2-Ethylhexyl)phthalate	0.83	10	330	149
Di-n-octylphthalate	0.39	10	330	149
Benzo(b)fluoranthene	0.19	10	330	252
Benzo(k)fluoranthene	0.30	10	330	252
Benzo(a)pyrene	0.21	10	330	252
Indeno(1,2,3-cd)pyrene	0.33	10	330	276
Dibenz(a,h)anthracene	0.28	10	330	278
Benzo(g,h,i)perylene	0.40	10	330	276
Additional analytes				
1,4-Dioxane	3 67	10	330	88
Pyridine	0.46	10	330	79
N-Nitrodimethylamine	0.51	10	330	42
Methyl methacrylate	3.36	10	330	69
Ethyl methyacrylate	2.16	10	330	69
2-Picoline	1.77	10	330	93
N-Nitrosomethylethylamine	1.51	10	330	88
Methyl methanesulfonate	1.33	10	330	80
N-Nitrosodiethylamine	1.37	10	330	102

Appendix A (Continued)
DETECTION LIMITS
for BNAs

Compound	ENCHEM[a] Method Detection Limit (µg/L)	ENCHEM[b] Reporting Limit Water (µg/L)	Solid (µg/kg)	1° Ion
Ethyl methanesulfonate	0 73	10	330	79
Pentachloroethane	2.70	10	330	117
Aniline	1.68	10	330	93
Benzyl alcohol	0.92	10	330	108
o-Toluidine	1.17	10	330	106
N-Nitrosopyrrolidine	2 02	10	330	100
Acetophenone	0 67	10	330	105
N-Nitrosomorpholine	1.41	10	330	56
3-Methylphenol	0 77	10	330	107
N-Nitrosopiperidine	1.17	10	330	114
a,a-Dimethylphenethylamine	2 13	20	670	58
o,o,o-Triethylphosphorothioate	2.23	10	330	198
Hexachloropropene	0.91	20	670	213
2,6-Dichlorophenol	0.78	10	330	162
N-Nitrosodi-n-butylamine	0.91	10	330	84
p-Phenylenediamine	18.5	100	3300	108
Safrole	1.37	10	330	162
Isosafrole	1.92	10	330	162
1,2,4,5-Tetrachlorobenzene	0.82	10	330	216
1,4-Naphthoquinone	1.80	10	330	158
1,3-Dinitrobenzene	1.41	10	330	168
Pentachlorobenzene	1 13	10	330	250
1-Naphthylamine	1.44	10	330	143
2-Naphthylamine	1.36	10	330	143
2,3,4,6-Tetrachlorophenol	2.28	10	330	232
5-Nitro-o-toluidine	0.96	10	330	152
Diphenylamine	0.79	10	330	169
1,3,5-Trinitrobenzene	0.96	10	330	213
Phenacetine	1.43	10	330	108
Diallate	2.91	10	330	86
4-Aminobiphenyl	0.68	10	330	169
Pronamide	1.59	10	330	173
Pentachloronitrobenzene	0 74	10	330	237
4-Nitroquinoline-1-oxide	2.61	10	330	190
Dinoseb	1.35	10	330	211
Methapyrilene	15.9	10	330	97
Aramite	4.27	20	670	185
p-Dimethylamino azobenzene	1.02	10	330	225

Appendix A (Continued)
DETECTION LIMITS
for BNAs

	ENCHEM[a] Method Detection Limit	ENCHEM[b] Reporting Limit		
		Water	Solid	1°
Compound	(µg/L)	(µg/L)	(µg/kg)	Ion
Chlorobenzilate	0 75	10	330	251
3,3-Dimethylbenzidine	2.65	10	330	212
2-Acetylaminofluorene	0 89	10	330	181
7,12-Dimethylbenz(a)anthracene	2.32	10	330	256
Hexachlorophene	38.2	500	17000	196
3-Methylcholanthrene	1.35	10	330	268
Benzidine	1 47	50	1700	184
1,2-Diphenylhydrazine	1.03	10	330	77
Benzoic acid	4.30	50	1700	122
Kepone	23.3	50	1700	272

[a] Method Detection Limit determination, USEPA 40CFR Pt.136, App.B, 1988. Method detection limits are updated periodically, the values currently in use may differ slightly from those published

[b] EN CHEM Reporting Limits based on internal Method Detection Limit determinations, USEPA 40CFR Pt.136, App.B, 1988.

Appendix B

Internal Standards

- 1,4-Dichlorobenzene-d4
- Naphthalene-d8
- Acenaphthene-d10
- Phenanthrene-d10
- Chrysene-d12
- Perylene-d12

Surrogate Standards

- 2-Fluorophenol
- Phenol-d6
- Nitrobenzene-d5
- 2-Fluorobiphenyl
- 2,4,6-Tribromophenol
- Terphenyl-d14
- 1,2-Dichlorobenzene-d4
- 2-Chlorophenol-d4

GC/MS Tuning Standard

- Decafluorotriphenylphosphine (DFTPP)

Appendix C

Matrix spike standard

The spike mixture contains the following components at 100 ug/mL:

Pyridine
N-Nitrosodimethylamine
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Benzyl alcohol
1,2-Dichlorobenzene
2-Methylphenol
2,2-oxybis(1-Chloropropane)
4-Methylphenol
Hexachloroethane
N-Nitroso-di-n-propylamine
Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
Benzoic acid
1,2,4-Trichlorobenzene
Naphthalene
4-Chloroaniline
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
1-Methylnaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
2-Chloronaphthalene
2-Nitroaniline
Dimethylphthalate
Acenaphthylene
2,6-Dinitrotoluene
3-Nitroaniline

Appendix C (continued)

Matrix spike standard (continued)

Acenaphthene
2,4-Dinitrophenol
Dibenzofuran
4-Nitrophenol
2,4-Dinitrotoluene
Diethylphthalate
Fluorene
4-Chlorophenyl phenyl ether
4-Nitroaniline
4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
1,2-Diphenylhydrazine
4-Bromophenyl phenyl ether
Hexachlorobenzene
Pentachlorophenol
Phenanthrene
Anthracene
Carbazole
Benzidine
di-n-Butylphthalate
Fluoranthene
Pyrene
Butylbenzylphthalate
Benzo(a)anthracene
3,3'-Dichlorobenzidine
Chrysene
bis(2-Ethylhexyl)phthalate
di-n-Octylphthalate
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene

Appendix D

GC/MS Tuning Criteria[1]
DFTPP

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	25-75% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 0 75% of mass 198
441	Present but less than 443
442	40-110% of mass 198
443	15-24% of mass 442

Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine[2]	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

System Performance Check Compounds (SPCC)

N-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

[1] follows CLP-SOW criteria.
[2] cannot be separated from N-Nitrosodiphenylamine .

Appendix E

SAMPLE ANALYSES
QUALITY CONTROL LIMITS[a]

Surrogate Compounds	Water %Rec.	Solids %Rec.	TCLP %Rec.
2-Fluorophenol	(21-79)	(47-98)	(33-72)
Phenol-d5	(18-47)	(48-99)	(19-49)
2-Chlorophenol-d4	(32-99)	(14-95)	(32-99)
1,2-Dichlorobenzene-d4	(54-115)	(33-104)	(64-106)
Nitrobenzene-d5	(57-115)	(43-101)	(63-109)
2-Fluorobiphenyl	(53-131)	(49-106)	(67-109)
2,4,6-Tribromophenol	(29-148)	(34-113)	(56-119)
Terphenyl-d14	(30-151)	(50-114)	(51-123)

[a] Control limits are updated periodically, the values currently in use may differ slightly from those shown above. Control limits are based on a mean value ± 3SD.

Appendix F

SAMPLE MATRIX SPIKE ANALYSES
QUALITY CONTROL LIMITS[a]

Matrix Spike Compounds	Water %Rec.	Solids %Rec.
Pyridine	(19-82)	(25-84)
N-Nitrosodimethylamine	(29-96)	(45-102)
Phenol	(22-60)	(52-104)
bis(2-Chloroethyl)ether	(68-106)	(47-105)
2-Chlorophenol	(72-96)	(48-105)
1,3-Dichlorobenzene	(73-94)	(44-100)
1,4-Dichlorobenzene	(72-96)	(46-100)
Benzyl alcohol	(67-89)	(50-106)
1,2-Dichlorobenzene	(72-99)	(46-103)
2-Methylphenol	(65-89)	(50-105)
2,2-oxybis(1-Chloropropane)	(65-104)	(41-114)
4-Methylphenol	(57-85)	(52-105)
Hexachloroethane	(63-106)	(48-102)
N-Nitroso-di-n-propylamine	(71-106)	(52-104)
Nitrobenzene	(66-111)	(51-106)
Isophorone	(67-107)	(42-113)
2-Nitrophenol	(77-100)	(51-104)
2,4-Dimethylphenol	(58-109)	(46-97)
bis(2-Chloroethoxy)methane	(75-102)	(50-109)
2,4-Dichlorophenol	(73-103)	(51-110)
Benzoic acid	(5-51)	(9-104)
1,2,4-Trichlorobenzene	(70-104)	(48-105)
Naphthalene	(73-104)	(54-103)
4-Chloroaniline	(76-111)	(13-53)
Hexachlorobutadiene	(57-120)	(47-112)
4-Chloro-3-methylphenol	(68-108)	(54-115)
2-Methylnaphthalene	(73-106)	(54-105)
1-Methylnaphthalene	(73-104)	(51-106)
Hexachlorocyclopentadiene	(31-142)	(3-130)
2,4,6-Trichlorophenol	(77-104)	(54-114)
2,4,5-Trichlorophenol	(78-104)	(55-116)
2-Chloronaphthalene	(79-101)	(55-109)
2-Nitroaniline	(67-114)	(53-122)
Dimethylphthalate	(79-103)	(56-117)
Acenaphthylene	(80-103)	(58-111)
2,6-Dinitrotoluene	(76-107)	(57-113)
3-Nitroaniline	(85-108)	(33-73)

Appendix F(Continued)

SAMPLE MATRIX SPIKE ANALYSES
QUALITY CONTROL LIMITS[a]

Matrix Spike Compounds	Water %Rec.	Solids %Rec.
Acenaphthene	(81-101)	(56-111)
2,4-Dinitrophenol	(60-116)	(27-93)
Dibenzofuran	(74-104)	(55-113)
4-Nitrophenol	(11-78)	(48-143)
2,4-Dinitrotoluene	(78-102)	(56-113)
Diethylphthalate	(76-106)	(54-117)
Fluorene	(76-115)	(56-119)
4-Chlorophenyl phenyl ether	(71-122)	(55-121)
4-Nitroaniline	(80-141)	(55-125)
4,6-Dinitro-2-methylphenol	(68-116)	(45-107)
N-Nitrosodiphenylamine	(76-119)	(55-126)
1,2-Diphenylhydrazine	(28-139)	(47-129)
4-Bromophenyl phenyl ether	(67-123)	(43-131)
Hexachlorobenzene	(63-125)	(37-137)
Pentachlorophenol	(63-127)	(51-104)
Phenanthrene	(79-107)	(59-117)
Anthracene	(79-108)	(53-114)
Carbazole	(89-158)	(79-149)
Benzidine	na	na
di-n-Butylphthalate	(74-113)	(53-122)
Fluoranthene	(78-110)	(56-122)
Pyrene	(79-111)	(61-115)
Butylbenzylphthalate	(75-112)	(55-116)
Benzo(a)anthracene	(70-125)	(58-113)
3,3'-Dichlorobenzidine	(29-172)	(18-94)
Chrysene	(76-116)	(54-132)
bis(2-Ethylhexyl)phthalate	(72-121)	(47-142)
di-n-Octylphthalate	(75-118)	(52-127)
Benzo(b)fluoranthene	(79-110)	(52-121)
Benzo(k)fluoranthene	(68-128)	(41-138)
Benzo(a)pyrene	(76-117)	(52-121)
Indeno(1,2,3-cd)pyrene	(72-127)	(54-135)
Dibenzo(a,h)anthracene	(68-134)	(51-138)
Benzo(g,h,i)perylene	(76-120)	(51-129)

[a] Control limits are updated periodically, the values currently in use may differ slightly from those shown above. Matrix Spike limits are based on a mean value ± 2SD.

na = not available

Appendix G

LABORATORY CONTROL SAMPLE ANALYSES
QUALITY CONTROL LIMITS[a]

LCS Compounds*	Water %Rec.	Solids %Rec.
Phenol	(21-68)	(54-123)
2-Chlorophenol	(44-154)	(50-128)
1,4-Dichlorobenzene	(67-99)	(46-109)
N-Nitroso-di-n-propylamine	(60-114)	(46-114)
1,2,4-Trichlorobenzene	(65-106)	(47-112)
4-Chloro-3-methylphenol	(48-157)	(58-132)
Acenaphthene	(67-113)	(64-108)
4-Nitrophenol	(15-76)	(54-159)
2,4-Dinitrotoluene	(74-107)	(65-110)
Pentachlorophenol	(42-161)	(47-140)
Pyrene	(63-129)	(69-112)

* The LCS spiking compound list may vary from above due to project requirements and scope. The QC control limits for other compounds not listed above will have advisory QC control limits until internal limits are developed.

[a] Control limits are updated periodically, the values currently in use may differ slightly from those shown above LCS Spike limits are based on a mean value ± 3SD

na = not available

Appendix H

TCLP LCS AND MS QUALITY CONTROL LIMITS[a]

Compounds	LCS %REC.	MS %REC.
Pyridine	(25-79)	(33-73)
2-Methylphenol	(49-109)	(52-96)
3-&4-Methylphenol	(86-196)	(29-104)
Hexachloroethane	(57-109)	(54-105)
Nitrobenzene	(70-116)	(69-111)
Hexachlorobutadiene	(61-109)	(44-112)
2,4,6-Trichlorophenol	(62-114)	(38-119)
2,4,5-Trichlorophenol	(53-120)	(62-113)
2,4-Dinitrotoluene	(65-108)	(48-134)
Hexachlorobenzene	(50-116)	(37-115)
Pentachlorophenol	(32-145)	(42-124)
Cresol, total	(10-155)	(15-126)

[a] Control limits are updated periodically, the values currently in use may differ slightly from those shown above. LCS Spike limits are based on a mean value ± 3SD and MS spike limits are based on a mean value ± 2SD.

Appendix I

DETERMINATION OF SPORADIC MARGINAL FAILURES ALLOWED

N[1]	X
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

N = Number of target analytes spiked.
X = Number of Sporadic Marginal Failures(SMF) allowed
[1] = The number of SMF allowances depend upon the number of target analytes reported from the analysis For instance, if the full list of target componds as presented in Appendix A are reported, then five (5) SMF's are allowed. If the Matrix Spike (MS) and/or the Laboratory Control Spike (LCS) includes only a subset of compounds and for surrogates, allow up to one (1) SMF for each B/N and A grouping. B = Base, N = Neutral, and A = Acid compounds.

NOTE: SMF's are used when QC limits have been established.

They are not used for compounds with advisory QC limits (i.e., QC limits have not yet been established).

ANALYTICAL METHOD

TITLE: Acid Digestion of Aqueous Samples for Total Metals - ICP

DEPARTMENT: Inorganic - Metals

APPLICATION: Samples may be analyzed by ICP for the following metals:

Al	Cd	Mg	Sn
As	Co	Mn	Se
Ba	Cr	Mo	Ti
Be	Cu	Na	Tl
B	Fe	Ni	V
Ca	K	Pb	Zn

Applicable to all aqueous matrices **except** Dissolved or Recoverable metals:
Reminder : This digestion is Not to be used for Ag, Sb, or Hg due to their volatility.

REFERENCE: EPA Manual SW-846, 3rd Edition, Method 3010A

PROCEDURE SUMMARY:

This method prepares samples for total metals analysis. Digestion can reduce interferences by organic matter and convert metals to a form that can be determined by instrumentation. The sample is heated with nitric acid and taken down to a low volume, then refluxed with nitric acid until the digestate is light or stable in color. Then refluxed with hydrochloric acid, cooled, and brought back to volume.

REVIEWED BY: Jeffrey A. Gordon — 2-29-00
Jeffrey A. Gordon
Inorganic Section Supervisor
Date

Gregory J. Graf — 2-28-2000
Gregory J. Graf
Quality Assurance Officer
Date

APPROVED BY: Glen A. Coder — 2-29-2000
Glen A. Coder
Laboratory Manager
Date

SAMPLE HANDLING AND PRESERVATION:

All samples are collected in plastic or glass containers. Samples are received by the laboratory acidified to a pH of < 2 with HNO_3 and stored at an ambient temperature.

INTERFERENCES:

The digestion is not suitable for samples to be analyzed by graphite furnace atomic absorption because hydrochloric acid can cause interferences during atomization.

APPARATUS AND MATERIALS:

Hot block
Acid repipetters
Pipette: adjustable and fixed volume
Hot block digestion tubes
Graduated cylinders: 50 mL
Hot block watchglasses
Whatman #541 filter paper
D.I. water bottle
Specimen cups: 120 mL

NOTE: All glassware is nitric acid (HNO_3) and DI water rinsed before use.

REAGENTS:

Deionized (D.I.) water
Nitric acid (HNO_3): concentrated, redistilled
Hydrochloric acid (HCl): 1:1

PROCEDURE:

<u>Quality Control</u>

Method Blank - one per digestion group, or per 20 samples, whichever is more frequent.

Laboratory Control Sample (LCS) - one per digestion group, or per 20 samples, whichever is more frequent.

Matrix Spike - one per digestion group, or per 20 samples, whichever is more frequent.
Matrix Spike Duplicate - one per digestion group, or per 20 samples, whichever is more frequent.

NOTE: For TCLP samples one MS is performed for each waste-stream. TCLP samples are spiked prior to preservation.

<u>Sample Preparation</u>

1. WEAR SAFETY GLASSES AND GLOVES.

2. Heat hot block to 90-95°C. Typically, setting the hot block at about 110 degrees will achieve the required temperature range. When stable, record temperature in daily log once per day.

3. Verify sample Station IDs and LIMs numbers on containers against LIMs worklists.

4. Lay out run in digestion log book. Identify QC samples at frequency stated above.

5. Prepare Laboratory Control Sample (LCS) by adding the appropriate LCS spiking solution to a 50 mL aliquot of D.I. water in a labeled digestion tube. See SOP MET-56

6. Prepare method blank by transferring a 50 mL aliquot of D.I. water into a labeled digestion tube using a graduated cylinder.

7. Transfer a 50 mL aliquot of well-mixed sample into a labeled digestion tube using a graduated cylinder. Do this for each sample in digestion group.

8. Spike designated samples using a calibrated pipettor. See SOP MET-56.

   CONTINUE DIGESTION IN HOOD.

9. Add 1.5 mL concentrated redistilled HNO_3 using a calibrated repipettor.

10. Swirl gently to mix.

11. Heat in 90-95°C hot block, slowly evaporating to 20-25 mL.

   CAUTION: DO NOT BOIL. DO NOT ALLOW TO GO DRY. IF SAMPLE GOES TO DRYNESS, DISCARD AND REPREPARE.

12. Remove from hot block and cool in hood.

13. Add 1.5 mL concentrated redistilled HNO_3 using a calibrated repipettor.

14. Cover with a watchglass and swirl gently to mix.

15. Continue heating, until digestion is light in color or does not change in appearance. (15 minutes)

   CAUTION: DO NOT ALLOW TO GO DRY.

16. Remove from hot plate and cool in hood.

17. Add 5 mL 1:1 HCl using a calibrated repipettor.

18. Cover again with watchglass and swirl gently to mix.

19. Heat 15 minutes to dissolve any residue.

20. Remove from hot block and cool in hood. Transfer to counter.

21. Rinse watchglass with D.I. water into digestion tube.

22. Filter samples with insoluble particles through Whatman #541 filter paper or equivalent into a labeled 120 mL specimen cup. OR use hot block digestion in-tube filters.

23. Bring to final volume of 50 mL.

24. Ready for analysis.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

INSTRUMENT TYPE: Inductively Coupled Plasma - Atomic Emission Spectroscopy

DEPARTMENT: Inorganic - Metals

APPLICATION: Digested sample matrices including water, soils, biota, industrial wastes, sludges, sediments, solid wastes, and leaching extracts for the determination of metals.

REFERENCES: EPA Method 200 7, EPA 600/4-91-010, June 1991
EPA Manual SW-846, 3rd Edition, Method 6010B

PROCEDURE SUMMARY:

Prior to analysis, samples are digested using appropriate sample preparation method (See EPA Digestion Methods 3005A, 3010A, 3050B, 3015, and 3051) Samples are introduced by a peristaltic pump, nebulized, and the resulting aerosol introduced to the plasma torch In the plasma, element specific atomic line emission spectra are produced The spectra are dispersed by a grating and the intensities of the lines are measured by a photomultiplier tube. Background correction is required for trace metal analysis. The background points selected may be on either or both sides of the analyte line of interest and must be in an area free of spectral interference.

Detection Limits:
See Attachment 1.

Range of Measurement:
See Attachment 1.

REVIEWED BY: Jeffrey A. Gordon — Date: 10-11-00
Jeffrey A. Gordon
Inorganic Section Supervisor

Gregory J. Graf — Date: 10-13-00
Gregory J. Graf
Quality Assurance Officer

APPROVED BY: Glen A. Coder — Date: 10-13-2000
Glen A. Coder
Laboratory Manager

SAMPLE HANDLING AND PREPARATION:

Aqueous samples are collected in plastic 250 mL, 500 mL, or 1000 mL bottles and preserved by adding HNO_3 to obtain a pH < 2. Once preserved, the sample holding time may not exceed 6 months Solid samples may be collected in plastic or glass containers and refrigerated at 4 °C.

INTERFERENCES:

Spectral interferences are caused by overlap of a spectral line from another element, background noise contribution, stray light from the line emission of high concentration elements and overlap from molecular band spectra. An interelement interference table (see current table), has been developed to help the analyst identify potential spectral interferences Selecting background correction points adjacent to the analyte line can compensate for stray light and background noise contribution. Physical interferences are effects associated with sample introduction and nebulization. Changes in viscosity, especially in samples with high dissolved solids and high acid concentrations, can cause significant inaccuracies during analysis.

APPARATUS:

- Thermo-Jarrell Ash 61E Trace, Inductively Coupled Argon Plasma Spectrophotometer
- 50 mL, 100 mL and 200 mL volumetric flasks
- Pipettes, adjustable and fixed
- 20 mL disposable beakers

REAGENTS

- Hydrochloric Acid (HCL), trace metal grade.

- Nitric Acid (HNO_3), redistilled.

- Mixed Calibration Standard: Calibration standards are prepared from High Purity (vendor) mixed and single element stock standards.

- Mixed Calibration Verification Standard (ICV, CCV): These standards are prepared from Inorganic Ventures (vendor) mixed and single element stock standards. These solutions must be prepared from a second, independent NIST traceable source

- Calibration Standards and/or Calibration Verification Standards that are of a concentration < 1 part per million will need to be prepared daily. Concentrations > 1 part per million will need to be made up weekly. Stock Standards are valid for one year as is the case with Reagents used during analysis.

- Single Element Stock Standards: Purchased from outside vendors and traceable to NIST SRMs. Certificates are kept on file.

- Mixed Stock Standards: Purchased from outside vendors and traceable to NIST SRMs. Certificates kept on file.

Calibration Blanks. Prepared at a concentration of 5% HCl and 5% HNO_3 with Milli-Q water

- Internal Standard: Prepared as per SOP Met-42. The internal standard is used in lieu of matrix matching.

SAMPLE PREPARATION:

Samples are prepared for ICP analysis using EPA analytical digestion methods. For recoverable metals see Method 3005A. For total metals see Method 3010A or 3015. For solid samples see Method 3050B or 3051. Dissolved metals do not need digestion if they were field filtered and acidified at the time of collection.

SAMPLE ANALYSIS:

Proceed with sample analysis by following EN CHEM SOP, "Inductively Coupled Plasma Emission Spectrometer" (2-MET-42).

NOTE: Any aqueous sample that is analyzed for selenium and is determined to have a detectable concentration present must be confirmed by GFAA. The only exception is TCLP extraction.

QUALITY CONTROL:

Initial Calibration Verification (ICV)
The ICV must be analyzed immediately after calibration and meet the rejection criteria of $\pm$ 10 % of the true value. Also, the relative standard deviation must be < 5 % from replicate (minimum of two) integrations. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value of the ICB must be $\leq$ 3 x IDL. For some analytes, i.e., Ba, Be, and Mn, the limit may be adjusted to 1/10 of the EQL. In cases where 3 x the IDL criteria is consistently exceeded, the QC officer may determine a limit from historical data. Recalibrate if the ICB fails. However, if the ICB concentration is < 1/10 the concentration of the associated samples, the analysis need not be terminated. Current control limits for ICB/CCB are established periodically and maintained on file in the laboratory

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. Rejection criteria is $\pm$ 10 % of true value. The relative standard deviation must be < 5% from replicate (minimum of two) integrations. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve. For ICP analysis, as long as the CCVs that bracket the samples to be reported for the analytes of interest are within the acceptable limit, the run is acceptable.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value of the CCB must be $\leq$ 3 x IDL. For some analytes, i.e., Ba, Be, and Mn, the limit may be adjusted to 1/10 of the EQL In cases where 3 x the IDL criteria is consistently exceeded, the QC officer may determine a limit from historical data. Recalibrate if the CCB fails. However, if the CCB is < 1/10 the concentration of

the associated samples, analysis need not be terminated For ICP analysis, as long as the CCBs that bracket the samples to be reported for the analytes of interest are within the acceptable limit, the run is acceptable.

Laboratory Control Sample (LCS)
The LCS is carried through all preparation procedures and analyzed for each matrix type with a frequency of 5 %. See current QC Charts for control ranges. In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be reprepared and/or reanalyzed.

Method Blank (MB)
A MB is carried through all prep procedures and analyzed with a frequency of 5 % Rejection criteria is < LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

Serial Dilution Test (For ICP analysis)
A Serial Dilution Test is performed if the analyte concentration is sufficiently high (50 X the EQL). For these samples a 1:5 dilution is performed and the result should agree within ± 10 % of the original value If the results do not agree the data is flagged with an [E flag.

Batch Post Spike (For ICP analysis)
A Batch Post Spike is required at a frequency of 5 %. The control limits for a post-spike are 75-125 %. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample, or use a higher concentration for the post spike that is more appropriate for the sample. Dilute appropriately until an acceptable recovery is obtained.

ICSA / ICSAB (For ICP analysis)
There is no criteria established for the ICSA; however, the analyst should be aware that if the result is greater than ± EQL, the IECs need adjustment. The criteria for the recoveries on the ICSAB are 80-120 %. There is no criteria on the minerals or for Fe or Al, as these elements are used as the interferent.

ACCURACY

One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5 %. Both QC samples must be calculated for accuracy. See current QC charts for control range.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with an [N qualifier. The control limits for a post-spike are 75-125%. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained. If only the matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding parent sample is flagged with an [MS qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however, the [N qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and LCS DUP must be used in its place.

PRECISION

Matrix spike duplicate samples are analyzed 1 per batch or at a frequency of 5 %, for samples that are similar in matrix.

For matrix spike duplicate samples, relative percent difference (RPD) is used to calculate compliance. See current QC charts for control limits.

Calculation:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with an [* flag.

Sample Result Calculations:

Aqueous Sample Calculation:

- Raw Data result (µg/L) X DF(also includes MW DF when applicable) = Final Result (µg/L)

Soil Sample Calculation:

- $$\frac{\text{Raw Data result (µg/L)} \times \text{FV (L)} \times \text{DF}}{\text{Sample Weight (g)} \times \text{Dry Weight (decimal form)}} = \text{Final Result (mg/kg dry weight corrected)}$$

Where: DF = Dilution Factor
MW = Microwave
FV = Final Volume

IF METHOD EPA 200.7 IS REQUESTED

For Boron analysis: use only plastic/Teflon containers from the time of collection.

Silver by this method is allowable up to 0.1 mg/L: dilute prior to digestion to result in < 0.1 mg/L in the analysis solution.

If barium is requested, samples with unknown SO4 concentrations should be analyzed ASAP

A matrix spike addition of 20-100 X the MDL should result in 90-110 % recovery or be within the established control limits.

Store calibration standards in plastic.

Samples which were preserved in house must be held for 16 hours prior to analysis.

ICV control limits are 95-105%.

If digestion is required: 1 LRB per batch at the time of analysis, 1 LFB per batch at the time of analysis.

If the analyte of interest is 90 % of its MCL or above, and the magnesium + calcium concentration is > 500 mg/L, use the method of standard additions.

Perform a matrix spike at a minimum of 10% of the samples or one per sample batch, whichever is greater.

SAFETY.

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and the Hazardous Waste Contingency Plan.

ATTACHMENT 1

Method Analytes and Reporting Limits

Analyte	EQL Water µg/L	EQL Soil/Biota µg/L	Linear Range
Silver	5.0	0.50	10,000
Aluminum	200	20	50,000
Arsenic	10	1.0	500,000
Boron	100	0 50	10,000
Barium	5.0	0.5	10,000
Beryllium	1 0	0.10	10,000
Calcium	100	50	500,000
Cadmium	1 0	0 10	50,000
Chromium	3 0	0.30	50,000
Copper	10	2.0	50,000
Cobalt	3 0	0 50	50,000
Iron - 259.9	50	10	50,000
Potassium - 766.4	100	100/500	50,000
Magnesium	30	30/50	500,000
Manganese	2 0	0.20/0.50	30,000
Molybdenum	10	1.0	50,000
Sodium - 330.2	500	200/1000	200,000
Nickel	5.0	0 50	50,000
Lead	5.0	0 50	50,000
Antimony	10	1.0	50,000
Selenium	10	1.0	50,000
Thallium	10	1.0	50,000
Tin	20	5.0	50,000
Titanium	20	2.0	50,000
Vanadium	5.0	0.50	50,000
Zinc	20	2.0/5 0	50,000

** Control limits and Linear Ranges are updated periodically. Those that are in use at the time of analysis will be used and made available to data validators.

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE: Alkalinity, Titrimetric, pH 8.3 and pH 4.5

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Drinking, surface and saline waters; domestic and industrial wastes.

REFERENCES: Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, Method 2320B

PROCEDURE SUMMARY:

Alkalinity of a water is its acid-neutralizing capacity. Alkalinity is a measure of an aggregate property of water and can be interpreted in terms of specific substances when it titrated to different end points These pH end points, indicate a function of carbonate, bicarbonate and hydroxide content in the water. A sample is titrated first to a pH of 8.3, then to a final pH of 4.5.

REPORTING LIMIT: 10 mg/L

REVIEWED BY: Jeffrey A. Gordon
Inorganic Section Supervisor
Date 10-11-00

Gregory J. Graf
Quality Assurance Officer
Date 10-13-00

APPROVED BY: Glen A. Coder
Laboratory Manager
Date 10-13-2000

SAMPLE HANDLING AND PRESERVATION:

The sample is collected in either a plastic or glass container and refrigerated at 4° C The holding time is 14 days from sample collection.

INTERFERENCES:

Substances such as salts of weak organic and inorganic acids in large amounts may cause interference.

Soaps, oily matter, suspended solids, or precipitates may coat the glass electrode and cause a sluggish response. Allow additional time between titration additions to let the electrode come to equilibrium and clean the electrode periodically or as required.

APPARATUS AND MATERIALS:

pH meter with glass electrode and automatic temperature compensation probe that can be read to 0 05 pH units
Specimen cups
Magnetic stir plate
Magnetic stir bars
Pipettes, glass
Volumetric flasks: 100 mL, 1 L
Buret: borosilicate glass, 50 mL
Graduated cylinders: plastic, 50 mL
Analytical balance
APG reference solution

REAGENTS:

Deionized (D.I.) water
Standard sulfuric Acid (H_2SO_4), 0.02 N, purchased commercially. Shelf Life = 1 year
Standard sulfuric Acid (H_2SO_4), 0.10 N, purchased commercially. Shelf Life = 1 year.
Sodium carbonate (Na_2CO_3) anhydrous, primary standard grade. Shelf Life = 1 year.

Prepare sodium carbonate stock standard, 10000 mg/L, as follows:

In a 1 L volumetric flask, dissolve 10.60 grams Na_2CO_3 in about 900 mL D.I water. Dilute to the mark and invert 3 times. Refrigerate. Shelf life = 1 year.

Prepare sodium carbonate working standards as follows.

Pipet 5.0 mL and 1.0 mL of 10000 mg/L sodium carbonate into separate 100-mL volumetric flasks.

Dilute each to the mark and invert 3 times. Label as 500 mg/L and 100 mg/L Na_2CO_3, respectively. Refrigerate. Shelf life = 1 week.

Prepare sodium carbonate standards for 0.10 N H_2SO_4 titration as follows·

Pipet 10 and 25 mL of 10,000 ppm sodium carbonate into two separate 100 mL volumetric flasks

Dilute to mark and invert three times. Label as 1000 and 2500 mg/L Refrigerate. Shelf life is 1 year.

PROCEDURE:

1. Calibrate pH meter according to EN CHEM SOP.
2. Use a graduated cylinder to place 50 mL of sample into a specimen cup
3. Add a stir bar and place on a stir plate
4. Measure the pH of the sample and record it on the bench sheet.
5. Titrate all samples initially with 0 02 N H_2SO_4 Add standard acid to the buret Record initial volume of buret. Titrate being careful to stir thoroughly but gently to allow meter to obtain equilibrium to 8.3.
6. Record volume of titrant used.
7. Titrate to a final pH of 4.5. Do Not refill the buret when going from pH 8.3 to pH 4.5. Record the total volume of titrant used.
8. For samples exceeding a concentration of 1000 mg/L, take a fresh aliquot of sample and titrate with 0.10 N H_2SO_4.

QUALITY CONTROL:

Initial Check Standards
Two standards are titrated at the beginning of each analysis day. A 100 mg/L and 500 mg/L are used when titrating with 0.02 N H_2SO_4 and 1000 mg/L and 2500 mg/L standards are used when titrating with 0.10 N H_2SO_4.

Laboratory Control Sample (LCS)
An LCS reference is purchased from APG as a setpoint standard. This standard must be analyzed with each batch or per 20 samples whichever is more frequent. The current control limits** are 81-115%. If the LCS does not meet current control limits, terminate analysis. Check validity of reagents, normality of acids, and/or pH calibration.

Method Blank
Place 50 mL of D.I. water into a specimen cup and add a stir bar Follow steps 3 through 7. A MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <LOD Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

Check standard
A check standard from a second source sodium carbonate stock is analyzed after every 10 analytical samples. Recovery must be ±10% of true value. If not, check pH calibration and repeat preceding 10 samples.

ACCURACY

One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. The current control limits** are 65-137%.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with a [N qualifier. The control limits for a post-spike are 75-125%

If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained. If only the matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding parent sample is flagged with an [MS qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however the [N qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and LCS DUP must be used in its place

To perform the MS and MSD spiking: add 5 mL of the 1000 mg/L working standard to 50 mL of sample. This will result in a 100 mg/L spike.

PRECISION

Matrix spike duplicate samples are analyzed 1 per batch or at a frequency of 5%, for samples that are similar in matrix.

Relative percent difference (RPD) is used to calculate matrix spike duplicate compliance. The current control limits** are 0-7%.

Calculation:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with an [* flag.

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to data validators

SAMPLE CALCULATIONS:

"Phenolphthalein Alkalinity" is the term traditionally used for the quantity measured by the titration to pH 8.3, irrespective of the colored indicator, if any, used in the determination

Potentiometric titration to an endpoint pH (8.3 and 4.5)

$$\text{Alkalinity, mg } CaCO_3/L = \frac{A \times N \times 50{,}000}{\text{Sample volume, mL}}$$

A = Volume of titrant, mL
N = Normality of standard acid

Calculation of Alkalinity Relationships

The results obtained from the phenolphthalein (pH 8 3) and total (pH 4.5) alkalinity determinations offer a means for stoichiometric classification of the three principle forms of alkalinity present in many waters. The classification ascribes the entire alkalinity to bicarbonate, carbonate and hydroxide according to this scheme:

1. Carbonate (CO_3^{2-}) alkalinity is present when pheno alkalinity is not zero but is less than total alkalinity.

2. Hydroxide (OH^-) alkalinity is present if pheno alkalinity is more than half of the total alkalinity.

3. Bicarbonate (HCO_3^-) alkalinity is present if the pheno alkalinity is less than half of the total alkalinity.

These relationships may be calculated using the following chart:

Result of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Concentration as $CaCO_3$
P = 0	0	0	T
P <½T	0	2P	T - 2P
P = ½T	0	2P	0
P >½T	2P - T	2(T - P)	0
P = T	T	0	0

SET No: 1

ANALYTICAL METHOD

TITLE: Nitrogen, Ammonia, Distillation Procedure

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Drinking, surface and saline waters, domestic and industrial wastes

REFERENCE: EPA 600 4-79-020, Revised March 1983, Method 350.1

PROCEDURE SUMMARY:

The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds. It is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by the LACHAT autoanalyzer or by ion specific electrode. All samples must be distilled prior to analysis.

REVIEWED BY: Jeffrey A. Gordon — 4-13-00 Date
Jeffrey A. Gordon
Inorganic Section Supervisor

Gregory J. Graf — 4-13-00 Date
Gregory Graf
Quality Control Officer

APPROVED BY: Glen A. Coder — 4-14-00 Date
Glen A. Coder
Laboratory Manager

SAMPLE HANDLING AND PRESERVATION:

Preserve samples with concentrated sulfuric acid, 2 mL per liter, and store at 4°C.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

APPARATUS AND MATERIALS:

Distillation apparatus: All glass, MIDI-Distillation system.
Tygon tubing: 3/8 x 1/16, 26 mm long
Collection Tubes
Graduated cylinders: 50 mL
pH meter
Beakers: 400 mL
Stir plate
Magnetic stir bar
Fixed Volume pipettor
Adjustable Volume pipettor
Boiling chips

INTERFERENCES:

Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile, alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation.

REAGENTS:

Deionized (D.I.) water, ammonia free
Ammonium chloride: NH_4Cl
Boric acid: H_3BO_3
Borate buffer
Sodium hydroxide: NaOH, 10N
Ammonia Free Water (Mili-Q water)
Sodium hydroxide: NaOH, 0.1N
Sodium Phenolate
Sodium Sulfite
APG reference solution for LCS
Sodium tetraborate, $Na_2B_4O_7 . 10\ H_2O$

NOTE: All solutions must be made with ammonia free water.

Prepare Ammonium chloride stock solution, NH4Cl, 1.0 mL = 1.0 mg NH_3-N (1000 ppm)

- Dissolve 3.819 grams NH_4Cl in D.I. ammonia free water and bring to volume in a 1 liter volumetric flask.
- Mix well. Shelf Life = 1 year.

Prepare Ammonium chloride working solution, 1.0 mL = 0.01 mg NH_3-N (10 ppm)

- Dilute 10 mL of stock solution to 1 liter in a volumetric flask using ammonia free D.I. water.
- Mix well. Shelf Life = 1 week.

Prepare Boric acid, H_3BO_3

- Dissolve 20 grams H_3BO_3 in ammonia free D.I. water.
- Dilute to 1 liter. Shelf Life = 1 year.

Prepare Borate buffer

- Add 88 mL of 0.1 N NaOH solution to 500 mL of 0.025M sodium tetraborate solution (9.5 grams anhydrous $Na_2B_4O_7 \cdot 10\ H_2O$ per liter) and dilute to 1 liter with D.I. water. Shelf Life = 1 year.

Prepare sodium hydroxide, NaOH, 1N

- Dissolve 40 grams NaOH in ammonia free D.I. water.
- Dilute to 1 liter. Shelf Life = 1 year.

Prepare sodium hydroxide, NaOH, 6N

- Dissolve 240 grams NaOH in ammonia free D.I. water.
- Dilute to 1 liter. Shelf Life = 1 year.

PROCEDURE:

1. Use a graduated cylinder to measure 50 mL sample, or aliquot of appropriate size, into a 400 mL beaker. Adjust to pH = 9.5 $\pm$ 0.1 with 10N NaOH, a dilution of 10N NaOH, or diluted H_2SO_4. For soils, weigh out 1.0 gram of the sample into a 400 mL beaker, add 50 mL of ammonia free water and adjust the pH as described above.

   NOTE: Do not adjust the pH more than 1 hour before distillation. Ammonia is very volatile.

2. Transfer the sample, pH adjusted, to the distillation tube. Add boiling chips. Add 2.5 mL borate buffer using a pipettor. Distill 40 mL into a 50 mL collection tube containing 6 mL boric acid.

NOTE: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution. Pull out the condenser tip before the heat has been turned off.

3. Dilute distillate to 50 mL with ammonia-free water.

4. Distill a Method Blank (MB) using 50 mL of ammonia free water per batch or every 20 samples whichever is more frequent.

5. Distill a Laboratory Control Sample (LCS) using 50 mL of the APG solution per batch or every 20 samples whichever is more frequent.

6. Distill a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) per batch or every 20 samples of a similar matrix whichever is more frequent. Use 0.1 mL of the ammonium chloride stock solution for the spiking solution. This should yield a final spiking concentration of 2.0 mg/L.

NOTE: If there is insufficient sample volume to perform an MS/MSD; perform an LCS/LCS DUP.

7. Transfer distillates to properly labeled 50 mL centrifuge tubes.

8. Store the distillates in the refrigerator until time of analysis. Analysis should be conducted within 5 days of the distillation. See EN CHEM SOP WCM-58 for the automated analysis procedure or EN CHEM SOP WCM-49 for the ion specific electrode analysis procedure.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE: Nitrogen, Ammonia – Automated Analysis

DEPARTMENT: Inorganic – Wet Chemistry

APPLICATION. Drinking, Surface and ground waters and Solid matricies

REFERENCE: EPA 600 4-79-020, Revised March 1983, Method 350.1

PROCEDURE SUMMARY.

This method is performed following distillation of the samples and is based on the Berthelot reaction Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration in the sample.

REPORTING LIMITS: Water. 0.10 mg/L; Soil: 25 mg/kg.

REVIEWED BY: ______________________ 10-11-00
Jeffrey A. Gordon — Date
Inorganic Section Supervisor

______________________ 10-13-00
Gregory J. Graf — Date
Quality Assurance Officer

APPROVED BY: ______________________ 10-13-2000
Glen Coder — Date
Laboratory Manager

SAMPLE HANDLING AND PRESERVATION:

Preserve samples with concentrated sulfuric acid, 2 mL per liter, and store at 4 °C. The maximum sample holding time prior to analysis is 28 days. ALL samples are distilled prior to analysis. For Distillation, see EN CHEM SOP WCM-25.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals

INTERFERENCES:

Calcium and magnesium ions may precipitate if present in sufficient concentration. Tartrate or EDTA is added to the sample in-line in order to prevent this problem. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration Sample color may be corrected for by running the samples through the manifold without color formation.

APPARATUS AND MATERIALS:

LACHAT Quikchem Automated Ion Analyzer
630 nm interference filter
75 cm sample loop
10-107-06-1-B ammonia manifold
Volumetric flasks: 100 mL, 1000 mL
Pipettor: adjustable, fixed

REAGENTS AND STANDARDS:

NOTE: All solutions must be made with ammonia free water.

Prepare Sodium Phenolate

<u>Caution: Wear gloves</u> Phenol causes severe burns and is rapidly absorbed into the body through the skin.

Dissolve 88 mL of 88% liquefied phenol or 83 g crystalline phenol (C_6H_5OH) in approximately 600 mL of DI water. While stirring, slowly add 32 g sodium hydroxide ($NaOH$). Cool, dilute to 1 L and invert to mix. Shelf life is 1 year.

Prepare Sodium Hypochlorite

In a 500 mL volumetric flask dilute 250 mL regular Clorox bleach to mark with D.I. water Shelf life is 1 year.

Prepare Buffer solution

Dissolve 50.0 g disodium ethylenediamine tetraacetate dihydrate ($Na_2EDTA\text{-}2H_2O$) and 9.0 g sodium hydroxide (NaOH) in approximately 900 mL D.I. water. Dilute to 1 L with D.I. water. Shelf life is 1 year

Prepare Sodium Nitroprusside

Dissolve 3.50 g of sodium nitroprusside (sodium nitroferricyanide [$Na_2Fe(CN)_5NO\text{-}2H_20$]) in approximately 900 mL of D.I water. Dilute to 1 L with D.I. water. Shelf life is 1 year.

Prepare Carrier and Diluent (0.20% Sulfuric Acid)

Add 2.0 mL concentrated sulfuric acid (H_2SO_4) to 900 mL of D I water. Dilute to 1 L with D.I water. Shelf life is 1 year.

Prepare Ammonium Chloride stock solution, NH_4Cl, 1.0 mL = 1.0 mg NH_3-N (1000 ppm).

Dissolve 3.819 grams NH_4Cl in approximately 900 mL of D.I. water. Dilute to 1 L with D.I. water. Shelf life is 1 year.

Prepare Ammonium chloride working solution, 10 mL = 0 10 mg NH_3-N (100 ppm).

Dilute 10 mL of stock solution to 100 mL in a volumetric flask with D.I. water. Prepare weekly.

Prepare Ammonium chloride calibration standards

Dilute 5.0, 3.75, 2.5, 1.25, 0 50, 0.10 mL of working solution to 100 mL in a volumetric flask with Diluent to make 5.0, 3.75, 2.5, 1.25, 0.50, 0.10 ppm standards respectively. Prepare 0.50, 0.10 ppm daily. Prepare 5.0, 3.75, 2.5, 1.25 ppm standards weekly.

Prepare Ammonium chloride check standard

Dilute 2.0 mL of 2nd source working solution to 100 mL in a volumetric flask with Diluent to make a 2.0 ppm standard. Prepare daily.

PROCEDURE:

1. Allow 15 min for heating unit to warm to 60° C.

2. Connect 10-107-06-1-B manifold.

3. Install 630 nm interference filter.

4. Install 75 cm sample loop.

CALIBRATION:

Instrument calibration is performed using the prepared standards at the following concentrations (mg/L): 0.10, 0.50, 1.25, 2.5, 3.75 and 5 0. Analyze the standards beginning with the highest and working to the lowest standard. The correlation coefficient for the curve must be 0.995 or greater The calibration must be verified as specified in Quality Control, below.

NOTE: All analyses on the LACHAT are performed using one replicate.

SYSTEM NOTES:

If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure

1 Place all reagent lines in deionized water and pump to clear reagents

2. Place all reagent lines in 1 M hydrochloric acid (1:12 concentrated HCL), pump for several minutes.

3. Place all reagent lines in deionized water and pump until the HCL is thoroughly washed out.

4. Resume pumping reagents.

QUALITY CONTROL·

- Correlation Coefficient (r value)
  The correlation coefficient, the measure of linearity of the standard curve, must be 0.995 or greater.

- Initial Calibration Verification (ICV)
  The ICV must be run immediately after calibration and meet 90-110% control limits. If not, recalibrate.

- Initial Calibration Blank (ICB)
  The ICB must be analyzed after the ICV and be less than the absolute value of the estimated quantitation limit (EQL). If not, recalibrate.

- Laboratory Control Sample (LCS) Distilled Standard
  The LCS is carried through all prep procedures and analyzed with a frequency of 5%. Recovery must meet the current control limit** of 72-116%

- Method Blank
  The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is < LOD. Other criteria may apply, such as regulatory limit and analyte concentration in samples.

- Continuing Calibration Verification (CCV)
  The CCV is analyzed after every 10 analytical samples and meet 90-110% control limits. If it is not within control, stop analysis, and recalibrate. All samples analyzed since the last successful CCV must be reanalyzed.

- Continuing Calibration Blank (CCB)
  The CCB is analyzed after every CCV and be less than the absolute value of the EQL. If it is not within control, stop analysis, and recalibrate. All samples analyzed since the last successful CCB must be reanalyzed

- Matrix Spike
  A spike must be performed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limit** of 80-114%

- Matrix Spike Duplicate
  A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits for accuracy and the difference between the MS and MSD must meet current control limits for precision. The current control limit** is 0-14% RPD

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to data validators.

CALCULATIONS:

Samples:

The instrument provides calculated sample results in mg/L, calculations are only necessary if a dilution was used.

Raw Data Value (mg/L) x Dilution Factor = Total Ammonia (mg/L)

Accuracy:

A matrix spike and matrix spike duplicate must be performed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for accuracy.

Spike calculation:

$$\% \text{ Recovery} = \frac{SSR-SR}{SA}$$

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP.

Precision:

A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for precision

Relative percent difference (RPD) calculation.

$$RPD = \frac{| MS-MSD |}{(MS+MSD)/2} \times 100$$

MS = Matrix Spike Value
MSD = Matrix Spike Duplicate Value

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE: Ion Chromatography

DEPARTMENT: Inorganic – Wet Chemistry

APPLICATION: Anions in drinking and surface waters, and groundwater.

PROCEDURE SUMMARY:

Method is used to determine Bromide, Fluoride, Chloride, Nitrite, Nitrate, Ortho-phosphate and Sulfate in ground water and surface water by use of an Ion Chromatograph.

REPORTING LIMIT:

See Attachment 1

REVIEWED BY: Jeffrey A. Gordon — Date: 10-11-00
Jeffrey A. Gordon
Inorganic Section Supervisor

Gregory J. Graf — Date: 10-13-00
Gregory J. Graf
Quality Assurance Officer

APPROVED BY: Glen A. Coder — Date: 10-13-2000
Glen A. Coder
Laboratory Manager

SAMPLE HANDLING AND PRESERVATION:

Sample can be collected in glass or plastic. Samples need to be unpreserved. Sample holding times: NO2, NO3, OPO4: 48 hours; F, CL, SO4: 28 days; Br: 28 days.

INTERFERENCES:

Samples that contain particles larger than 0.45 microns and reagents solutions that contain particles larger than 0.20 microns require filtration to prevent damage to the instrument column or flow system.

APPARATUS AND MATERIALS.

DX – 120 Ion Chromatograph
5.0 mL sample vials and filter caps
Volumetric flasks: 100 mL, 1000 mL
Pipettors adjustable and fixed volume (3.0, 1.5, 1.0, 0.10, 0.050, 0.010 mL)

REAGENTS AND STANDARDS:

NOTE All solutions must be made with Milli-Q® water.

Prepare Eluent:

Dilute 10 mL of concentrated eluent to 1 L with Milli-Q® water.

Prepare Anion Calibration Standards:

Dilute 3.0, 2.5, 1.5, 1.0, 0.10, and 0.020 mL of 1000 ppm APG Standard of each of the following anions (Br, F, CL, NO_2, NO_3, OPO_4, SO_4) in 100 mL volumetric with Milli-Q® water to make 30, 25, 15, 10, 1.0, 0.20 ppm standard solutions. Prepare standards weekly, except those < 1 ppm which are prepared daily.

Prepare Anion Check Standard:

Dilute 1.5 mL of 1000 ppm APG Standard of each of the following anions (Br, F, CL, NO_2, NO_3, OPO_4, SO_4) in 100 mL volumetric with Milli-Q® water to make a 15 ppm standard. Source of standard should be of a different lot than that of calibration standards. Prepare weekly.

PROCEDURE:

1. Execute PeakNet.

2. Click on "Run Method".

3 Open method file· "Startup".

4. Run manual baseline until stable, usually 10 minutes

5 Open "Schedule" from PeakNet main menu.

6. Type in analytical run you wish to perform:

   a. For calibration standards, be sure to include sample type as "calibration std" and level as, e.g.: Cal1= level 1, Cal2 = level 2.

   b ICV, ICB, CCV, CCB: Sample type is "check std". CCV, ICV = level 1 and CCB, ICB = level 2.

   c. All others need sample type "sample".

   d. Enter Method: En Chem Anions.met (Br, CL, F, NO_2, NO_3, OPO_4, SO_4)

   e. Enter data file: yymmdd.

   f. Save schedule: yymmdd

   g. After last CCB enter sample "shutdown" with Method. shutdown.met.

7. From run window: Load schedule:

   a. Click on mode tab.

   b. Click on "run via external signal".

8. Load autosampler.

   a. Pour 5 mL of sample into each vial.

   b. Seat filter cap so top of cap is even with top of vial.

9. Press run/hold button of autosampler.

10. Once analytical run has completed, go to PeakNet main menu and open "Optimize".

11. Each sample produces it's own .dat file and chromatograph. Open each file in the optimize window, check and correct peak naming and void volumes. Excess peaks can be deleted.

12. Once all samples have been optimized, open "Batch" from PeakNet main menu.

    a. Select input:

       1. Select schedule which you wish to batch.

       2. Lines used should be one less than total lines in schedule.

    b. Click output tab:

1. Click on summary report

c. Click export tab:

1. Type in name of download file· e g . G:\data\inorgani\ic\yymmdd

CALIBRATION AND STANDARDIZATION:

Perform calibration with standards at the following levels (ppm): 0.20, 1.0, 10, 15, 25 and 30 These standards define the linear range for the analysis. The correlation coefficient must be 0.995 or greater.

QUALITY CONTROL:

Correlation Coefficient (r-value)
The correlation coefficient, the measure of linearity of a standard curve, must be 0.995 or greater. If the value is less than 0.995, recalibrate the instrument

Initial Calibration Verification (ICV)
The ICV must be analyzed immediately after calibration and meet the rejection criteria of $\pm$ 10% of the true value. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

APG Check Standard
An APG check standard for each anion must be analyzed weekly to check column efficiency, accuracy, etc Rejection criteria is $\pm$ 10% of the true value. If the APG fails, the separation column will be replaced or taken out of service until it can be cleaned. Results will be recorded in the IC Daily Log.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be $\leq$ EQL. Recalibrate if it fails.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. Rejection criteria is $\pm$ 10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve As long as the CCVs that bracket the samples to be reported for the analytes of interest are within the acceptable limit, the run is acceptable

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be $\leq$ EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

Laboratory Control Sample (LCS)
The LCS is carried through all preparation procedures and analyzed for each matrix type with a frequency of 5%. See Attachment 1 for control ranges. In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be reprepared and reanalyzed.

Method Blank (MB)
The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

ACCURACY

One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. See Attachment 1 for control range.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with an [N qualifier.

The control limits for a post-spike are 75-125%. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained. If only the matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding parent sample is flagged with an [MS qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however, an [N qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and an LCS DUP must be used in its place.

PRECISION

Matrix spike duplicate samples are analyzed 1 per batch or at a frequency of 5%, for samples that are similar in matrix.

For matrix spike duplicate samples, relative percent difference (RPD) is used to calculate compliance. See Attachment 1 for control limits.

Calculation:

$$RPD = \frac{|MS - MSD|}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with an [* flag.

Sample Result Calculations:

Aqueous Sample Calculation:

Raw Data result (mg/L) X DF = Final Result (mg/L)

DF = Dilution Factor

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

ATTACHMENT 1

ANION	REPORTING Limit (mg/L)	LCS Control Limit (% REC)	MS/MSD Control Limit (% REC)	RPD Control Limit (MAX %)
Bromide	0.20	90-110	90-114	5
Fluoride	0.10	90-117	76-122	4
Chloride	2.0	88-111	76-113	7
Nitrate	0.20	90-110	90-110	4
Nitrite	0.20	90-110	90-110	4
Ortho-phosphorus	0.20	79-129	75-145	5

** Control limits are updated periodically The control limits that are in use at the time of analysis will be used and made available to data validators.

En Chem, Inc.

SET No: 1

Quality Assurance Document

ANALYTICAL METHOD

TITLE: DX-120 Ion Chromatograph -Instrument Operating Procedure

DEPARTMENT: Inorganic – Wet Chemistry

APPLICATION: DX-120 Ion Chromatograph

REFERENCE: DX-120 Ion Chromatography Operators Manual

PROCEDURE SUMMARY:

Method is used to determine Bromide, Fluoride, Chloride, Nitrite, Nitrate, Ortho-phos, Sulfate, in ground water and surface water by use of an Ion Chromatograph.

REVIEWED BY: Jeffrey A. Gordon
Jeffrey A. Gordon
Inorganic Section Supervisor

3-24-00
Date

Gregory J. Graf
Gregory J. Graf
Quality Assurance Officer

3-28-00
Date

APPROVED BY: Glen A. Coder
Glen Coder
Laboratory Manager

3-29-00
Date

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APPARATUS AND MATERIALS:

DX – 120 Ion Chromatograph
Concentrated eluent: Dionex AS14 Eluent Concentrate, Part # 53560, or equivalent.
Volumetric flasks: 1000mL
Pipettor: adjustable, fixed (5.0 mL)

REAGENTS AND STANDARDS:

NOTE: All solutions must be made with milli - Q water.

Prepare Eluent

Dilute 10 mL of concentrated eluent to 1 L with milli – Q water. Make fresh weekly.

PROCEDURE:

1. Fill eluent bottle with fresh eluent.
2. Execute PeakNet software.
3. From PeakNet main menu open "Run Method"
4. On DX-120 front control console:
    a. Press "local/remote" button to local setting.
    b. Press "eluent pressure".
    c. Open front door of DX-120.
    d. Turn transducer valve 1 ½ turns counter clockwise.
    e. Turn on pump from front control panel.
    f. Turn flow control knob until flow is 2.00 mL/min.
    g. After 10 seconds turn flow setting to 1.30 mL/min and close transducer valve.
    h. Turn on SRS from front control of panel.
    i. Close DX-120 front door.
5. Open method "startup.met".

a. Click on mode tab.

b. Click on "run from menu".

6. Click on "OPTIONS" on menu bar.
7. Click on "run baseline".
8. Allow unit to stabilize for ten minutes.
9. Instrument is now ready to analyze samples. (See EN CHEM SOP WCM-60)

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE: Chemical Oxygen Demand, Colorimetric, Manual (Vial - Low)

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Surface, Ground waters, domestic and industrial wastes

REFERENCE: EPA manual 600 4-79-020, March 1983, Method 410 4.

PROCEDURE SUMMARY:

Blanks and standards are sealed in tubes that are heated in an oven in the presence of dichromate at 150°C. After two hours, the tubes are removed, cooled and measured spectrophotometrically at 440 nm

REPORTING LIMIT. 10 mg/L

REVIEWED BY: G. Gordon
Jeffrey A. Gordon
Inorganic Section Supervisor
Date 10-11-00

Gregory J. Graf
Gregory J. Graf
Quality Assurance Officer
Date 10-13-00

APPROVED BY: Glen A. Coder
Glen A. Coder
Laboratory Manager
Date 10-13-2000

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SAMPLE HANDLING AND PRESERVATION:

Samples are collected in glass bottles and preserved with H_2SO_4 to a pH of 2 and cooled to 4° C. Holding Time = 28 days.

INTERFERENCES:

Chlorides are quantitatively oxidized by dichromate and represent a positive interference Mercuric sulfate is added to the digestion tubes to complex the chlorides.

APPARATUS AND MATERIALS:

10 mL sealed OIC standard level ampules which contain all reagents for the test
Mechanical ampule sealer capable of providing strong consistent seals
Oven capable of maintaining 150° C $\pm$ 2° C
Hach Spectrophotometer DR2000
Volumetric flasks: 50 mL, 1000 mL
Adjustable and Fixed Volume pipettors
Wire racks
Insulated gloves

REAGENTS:

Deionized (D.I.) water
Potassium acid pthalate

Prepare COD Stock Standard, 1.0 mg/L

Add 0.8503 grams of potassium acid phthalate to a 1 liter volumetric flask and dilute to the mark. (Shelf life = 1 year).

Prepare Calibration Standards

Prepare a series of standards using the stock standard as follows:

MilliLiters of COD Solution 1 mL = 1.0 mg COD	Standard Concentration when Diluted in 100 mL Volumetric Flasks mg COD/Liter
0.00	0.0
0.50	5.0
1 00	10 0
2.00	20.0
5 00	50.0
10.0	100.0
15.0	150.0

Optimum concentration:	20 -150 mg/L
Reporting Limit.	EQL = 10 mg/L

PROCEDURE

1. Unseal the OIC ampules.

2. Carefully pipet 2.5 mL sample using a calibrated adjustable pipettor into each ampule such that it forms a layer on top of the reagents contained in the ampule.

   NOTE: Samples containing particulates should be roughly homogenized and milled in a blender or similar device before adding the sample to the ampule

3 Carefully seal the ampule. It is recommended that a mechanical ampule sealer be used which is designed to form a strong, consistent seal. During digestion, the reagents and sample are raised to a point just below boiling. Improperly sealed ampules may leak or break. Ampules should be checked for leakage by running top of ampule over paper towels.

4. Thoroughly mix the contents of the sealed ampule by shaking.

   CAUTION: The ampule will get very hot during mixing. It is recommended that ampules be mixed either in racks or with the use of insulated gloves. Eye protection MUST be worn

5. Place the ampule in the oven at 150° C ± 2° C for 2 hours.

6. Invert ampule once to mix contents after removing from oven and allow to cool to room temperature. If rapid cooling is desired, the ampules may be placed in a water bath. If, however, certain samples form crystals, discontinue the rapid cooling and allow these samples to cool slowly in the room air.

7. Allow any suspended precipitate to settle for 10 minutes.

8. Set up the Hach spectrophotometer. The 150 mg/L standard should be used as a procedural blank to zero the spectrophotometer. After zeroing, use the remaining standards to prepare a procedural curve. Standard levels are (mg/L)· 150, 100, 50, 20, 10, 5 and 0.0. Read the absorbance of each ampule on the spectrophotometer at 440 nm wavelength. By use of the standard curve, the absorbance is converted graphically into mg/L\COD.

9. Samples which exceed 150 mg/L are analyzed using the mid-level COD method (En Chem SOP WCM-40).

Quality Control

Correlation Coefficient (r-value)
The correlation coefficient, the measure of linearity of a standard curve, must be 0.995 or greater. If the value is less than 0.995 recalibrate the instrument.

Initial Calibration Verification (ICV)
The ICV must be analyzed immediately after calibration and meet the rejection criteria of $\pm$10% of the true value. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be $\leq$EQL. Recalibrate if it fails.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. Rejection criteria is $\pm$10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be $\leq$EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

Laboratory Control Sample (LCS)
The LCS is carried through all preparation procedures (approximately 100 mg/L), and analyzed for each matrix type with a frequency of 5%. The current control limit** is 71-122%. In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be reprepared and reanalyzed.

Method Blank (MB)
A MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

ACCURACY

One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. The current control limits are 57-135%.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with an [N qualifier. The control limits for a post-spike are 75-125% If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained If only the matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding parent sample is flagged with an [MS qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however, an [N qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary

Spike with 1.0 mL of 100 ppm standard and 1.5 mL of sample

Calculation: $\dfrac{(2.5)\,(\text{spike value}) - (1.5)\,(\text{sample value})}{100\ \text{ppm}}$

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and an LCS DUP must be used in its place.

PRECISION

Matrix spike duplicate samples are analyzed 1 per batch or at a frequency of 5%, for samples that are similar in matrix.

For matrix spike duplicate samples, relative percent difference (RPD) is used to calculate compliance The current control limit** is 0-24%.

Calculation:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with an [* flag.

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to data validators.

Sample Result Calculations·

Aqueous Sample Calculation:

Enter the standard curve into a calculator with a linear regression program. Enter the absorbance of the samples to obtain the total mg of sample.

$$\text{COD mg/L} = \frac{2.5}{A} \times B \times C$$

Where: A = mL sample used
B = dilution factor, if any
C = mg COD from Standard Curve

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE:	MIDI-DIST Cyanide Distillation
DEPARTMENT:	Inorganic - Wet Chemistry
APPLICATION:	Drinking, surface, domestic and industrial waste, and soils
REFERENCE:	EPA 600, August 1993, Method 335.4 EPA Statement of Work, Method 335.4, CLP SOW ILM 4.0

PROCEDURE SUMMARY:

The cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with strong acid and distillation of the HCN into an absorber-scrubber containing sodium hydroxide solution. The cyanide ion concentration in the absorbing tube is then determined by automated colorimetry. All samples must be distilled prior to analysis.

REVIEWED BY: Jeffrey A. Gordon, Inorganic Section Supervisor — Date: 11-29-00

Gregory J. Graf, Quality Assurance Officer — Date: 11-29-00

APPROVED BY: Glen A. Coder, Laboratory Manager — Date: 11/29/2000

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SAMPLE HANDLING AND PRESERVATION:

Preserve samples with 10 N NaOH to pH >12 and store at 4°C. Sample Hold time is 14 days.

INTERFERENCES:

1. Chlorine: Test a drop of sample on KI paper - blue coloration indicates a positive result. Gradually add ascorbic acid until no blue color is obtained using KI paper.

2. Sulfides: If a drop of distillate darkens lead acetate paper, treat the sample with cadmium carbonate, yielding cadmium sulfide (yellow). Filter before distillation.

3. Nitrate/Nitrite: Some organic compounds decompose to form HCN. Add sulfamic acid to eliminate interference.

APPARATUS AND MATERIALS:

MIDI-VAC Distillation block
Distillation Tube
Cold Finger Condenser
Absorber Head
Tygon Tubing
Cooling water
Vacuum source
Spatula
Graduated cylinders - 25ml, 50ml
D.I. water
20% sulfuric acid

REAGENTS:

0.25N NaOH Solution: Dissolve 20g NaOH in D.I. water. Dilute to 2 liters
Cadmium Carbonate - Powdered
Ascorbic Acid
Sulfamic Acid
Sulfuric Acid - Concentrated
Magnesium Chloride: Dissolve 1020g $MgCl_2 \cdot 6H_2O$ in 1600ml D.I. water. Dilute to 2 liters.
Potassium Cyanide
Potassium Hydroxide
ERA reference standard

Stock Cyanide Solution: Dissolve 2.51g KCN and 2g KOH in 900ml D.I. water, dilute to one liter. Standardize Weekly with 0.0192 N $AgNO_3$.
NOTE: Use extreme caution when working with KCN: dissolve KCN in a hood.

Working Cyanide Solution: Dilute 5ml stock cyanide solution to 1000ml with D.I. water (1ml = 5.0μg CN). Add 2g NaOH. Shelf Life = 1 Week.

PROCEDURE:

1. Prepare Digestion Log for samples, 5% quality control is required.

2. Label distillation tubes with respective sample id.

3. For aqueous samples: Measure 50ml of sample into distillation tube. Add 2-3 boiling chips.
   For solid samples: Weigh 1.0g of sample (to the nearest 0.01g) into the distillation tube and dilute to 50ml with D.I. water. Add 2-3 boiling chips.

4. Add 50ml of 0.25N NaOH to the absorber tube.

5. Connect the distillation tube, condenser, and absorber in train.

6. Turn on main vacuum and adjust each sample position until three bubbles/second are observed in distillation tube.

7. Turn on cooling water to cold finger condensers. Flow meter should have a reading of 60 GPH before proceeding.

8. Add 0.2g of sulfamic acid through top air inlet tube of the distillation head. Mix for three minutes.

9. Add 5ml 18N sulfuric acid through air inlet tube. Rinse with D.I. water. Allow air flow to mix the tube contents for 5 minutes.

10. Add 2ml of magnesium chloride solution through air inlet tube. Rinse with D.I. water.

11. Set timer to 120 minutes and set distillation block to 124 °C. Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum flow.

12. Refluxing should occur for 90 minutes of the 120 minute total (at 70 °C).

13. Allow flask to cool for 20 minutes. Vacuum and cooling water should remain on.

14. Turn off vacuum pump and cooling water.

15. Transfer contents of absorber tubes into 50ml centrifuge tubes and store at 4 °C until analyzed. Analyze within 5 days of the distillation. (Keeping in mind the 14 day Hold time)

16. Distill a Method Blank (MB) using 50 mL of D.I water per batch or every 20 samples whichever is more frequent.

17. Distill a Laboratory Control Sample (LCS) per batch or every 20 samples whichever is more frequent.

    For Aqueous Samples: Use 5 mL of the cyanide working standard diluted to 250 mL. Then measure out 50 mL of this solution for the LCS. The concentration will be 0.1 mg/L.

    For Soils: Weigh 1.0 gram of the ERA reference standard into the distillation tube and add 50 mL of D.I. water. Also set-up the same 0.1 mg/L sample as described for the Aqueous samples LCS.

18. Distill a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) per batch or every 20 samples of a similar matrix whichever is more frequent. Use 1.0 mL of the cyanide working standard. This should yield a final spiking concentration 0.1 mg/L.

NOTE: If there is insufficient sample volume to perform an MS/MSD; perform an LCS/LCS DUP.

19. See EN CHEM SOP WCM-23 for the automated analysis procedure.

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE:	Total Cyanide, Automated Analysis
DEPARTMENT:	Inorganic - Wet Chemistry
APPLICATION:	Alkaline distillates of water, wastewater, and solids for total cyanide.
REFERENCE:	EPA 600 4-79-020, Revised March 1983, Method 335.4 EPA Manual SW-846, 3rd Edition, Method 9012A QuikChem Method No. 10-204-00-1-A

PROCEDURE SUMMARY:

The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a manual reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solutions. Distillation is described in SOP WCM-37. The cyanide ion in the absorbing solution is converted to cyanogen chloride by reactions with Chloramine-T, that subsequently reacts with pyridine and barbituric acid to give a red-color complex. The color is read at 570 nm.

REPORTING LIMITS: Water: 0.010 mg/L; Soil: 0.50 mg/kg

REVIEWED BY: ______________________ 11-29-00
Jeffrey A. Gordon — Date
Inorganic Section Supervisor

______________________ 11-29-00
Gregory J. Graf — Date
Quality Control Officer

APPROVED BY: ______________________ 11/29/2000
Glen A. Coder — Date
Laboratory Manager

SAMPLE HANDLING & PRESERVATION:

Distillates are preserved in 0.25 N sodium hydroxide (NaOH).

INTERFERENCES:

Most interferences are eliminated by distillation. Distillates can be checked for sulfides by placing 1-2 mL distillate onto $CdCO_3$, a yellow color being positive for sulfide interferences. If positive, the entire distillate is then treated with approximately 1 gram $CdCO_3$, mixed thoroughly and filtered. Samples should be checked prior to distillation for the presence of hypochlorite with KI starch paper. A blue/black color being positive. The sample is then treated by the gradual addition of sodium thiosulfate until the KI starch paper does not change color.

APPARATUS AND MATERIALS:

Lachat QuikChem Automated Ion Analyzer
Heater module
570 nm interference filter
150 cm sample loop
10-204-00-1-A Cyanide manifold
Volumetric flasks: 100 mL, 500 mL, 1000 mL
Pipettor
Burette
2 meter back pressure loop, 0.52 mm i.d.

REAGENTS:

Deionized (D.I.) water
Sodium hydroxide (NaOH)
Potassium phosphate, monobasic (KH_2PO_4)
Chloramine-T
Pyridine
Barbituric acid
Hydrochloric acid (HCl)
Helium gas
Potassium hydroxide
Potassium cyanide
Silver nitrate
p-dimethylaminobenzalrhodanine
Acetone

Prepare Carrier Solution, 0.25 N NaOH

- Dissolve 10 grams NaOH in 900 mL D.I. water. Dilute to 1 liter. Shelf Life is 1 year

Prepare Buffer Solution

- Dissolve 97 grams KH_2PO_4, potassium phosphate monobasic, anhydrous in approximately 800 mL D.I. water. Dilute to 1 liter. Shelf Life is 1 year.

Prepare Chloramine-T

- Dissolve 2 grams Chloramine-T hydrate in 400 mL D.I. water. Dilute to 500 mL. Prepare fresh daily.

Prepare Pyridine-Barbituric acid Reagent

- Make under hood! Place 15 grams barbituric acid in a 1 liter volumetric flask using approximately 100 mL D.I. water to rinse down the sides.
- Add 75 mL pyridine (C_5H_5N) and mix to dissolve the barbituric acid.
- Add 15 mL conc. HCL. Stir and dilute to 1 liter. Shelf Life is 1 year

STANDARD PREPARATION:

Prepare Indicator Solution

- Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 mL acetone. Shelf Life is 1 year

Prepare Stock Standard, 1000 mg/L

WARNING: DANGER! POISON! May be fatal if swallowed. Contact with acid liberates poisonous gas.

- Dissolve 2 grams potassium hydroxide (KOH) in approximately 500 mL D.I. water in a 1 liter volumetric flask.
- Add 2.51 grams potassium cyanide (KCN).

WARNING: AVOID INHALATION OF DUST OR CONTACT WITH SOLID OR SOLUTION OF KCN.

- Dissolve and dilute to 1 liter. Shelf Life is 1 year

Stock Standard verification

- The stock standard must be standardized weekly to verify the concentration. **This standardization must be documented in the logbook.**
- Standardize the stock standard against 0.01920 N silver nitrate ($AgNO_3$) at the time of preparation and on a weekly basis. Using a buret, titrate a 1.0 mL aliquot of the cyanide stock diluted to 100 ml standard with 0.25 N NaOH and 0.5 mL indicator solution, with $AgNO_3$ solution to a salmon colored endpoint. **Record the volume of titrant used in the logbook.**

- Perform the titration in triplicate along with a blank of 100 mL 0.25 N NaOH with 0.5 mL indicator solution. Subtract the volume of titrant used on the blank from the amount used to titrate the sample and calculate the concentration of the Stock Standard. **Record the concentration of the stock cyanide solution in the logbook.**
  1 mL $AgNO_3$ = 1 mg CN

Prepare Working Standard: 5.0 mg/L

- Dissolve 2 grams NaOH in approximately 500 mL D.I. water in a 1000 mL volumetric flask. Shelf Life is 1 week.

From the above determination of the stock standard concentration, use the following formula to determine the volume of stock standard solution needed to prepare the 5.0 mg/L working standard.

Note: If the concentration of the standard must be between 995 mg/L and 1005 mg/L, then no correction for concentration is needed. Use 5.0 ml of stock solution.

$$\text{Milliliters (ml) of stock std. to prepare 5.0 mg/L std} = \frac{5000}{\text{Stock Std Conc. (mg/L)}}$$

- Pipette required volume of stock standard into the flask and dilute to mark with D.I. water.

- Transfer to Storage bottle and record the preparation date on the label.

Prepare Calibration Standards (DAILY)

- Into 4 - 100 mL volumetric flasks, pipette 4.0, 2.0, 1.0 and 0.2 mL Working Standard Solution.

- Dilute to the mark with 0.25 N NaOH. This makes standards of 0.20, 0.10, 0.050 and 0.010 mg CN/L respectively. Use 0.25 N NaOH as the blank.

Prepare ICV/CCV (DAILY)

- Pipette 2.0 mL. of 5.0 mg/L working standard from the alternate source into 100 mL volumetric flask. Dilute to the mark with 0.25 N NaOH. This makes 0.1mg CN/L check standard.

Prepare LCS (0.1ppm) (DAILY)

- Pipette 1.0 mL of 5.0 mg/L working standard from the alternate source into a 50 mL midi-distillation apparatus.

NOTE: for LCS preparation and matrix spike additions, use a 1.0 mL class A pipette.

PROCEDURE:

1. Start up instrument. (See ENCHEM SOP WCM-29).
2. Connect the 10-204-00-1-A Cyanide Manifold.
3. Install 570 nm filter.
4. Install 150 cm sample loop.
5. Install back pressure loop.
6. Set heater at 60°C and allow 15 minutes for unit to warm up.
7. Place reagent lines into proper containers for collecting waste from cyanide analysis.
NOTE: All analyses on the LACHAT are performed using one replicate.

Calibration

The instrument must be calibrated every time it is set up. The working linear range is from 0.010 to 0.20 mg/L CN^-.

Quality Control

- Correlation Coefficient (r value)
The correlation coefficient, the measure of linearity of the standard curve, must be 0.995 or greater.

- Initial Calibration Verification (ICV)
The ICV must be run immediately after calibration and meet 90-110% control limits. If not, recalibrate.

- Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV and be less than the absolute value of the estimated quantitation limit (EQL). If not, recalibrate.

- Laboratory Control Sample (LCS): Distilled Standard
A 0.1 ppm standard must be distilled and analyzed for each 20 samples, and meet control limits of 90-110%.

- Method Blank

The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is < LOD. Other criteria may apply, such as regulatory limit and analyte concentration in samples.

- Continuing Calibration Verification (CCV)
  The CCV is analyzed after every 10 analytical samples and meet 90-110% control limits. If it is not within control, stop analysis, and recalibrate. All samples analyzed since the last successful CCV must be reanalyzed.

- Continuing Calibration Blank (CCB)
  The CCB is analyzed after every CCV and be less than the absolute value of the EQL. If it is not within control, stop analysis, and recalibrate. All samples analyzed since the last successful CCB must be reanalyzed.

- Matrix Spike
  A spike must be performed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits which are 68-137 for water, and 62-113 for soils**.

- Matrix Spike Duplicate
  A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits for accuracy and the RPD between the MS and MSD must meet current control limits for precision. Current limits are 14 for water and 23 for soils**.

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to the data validators.

CALCULATIONS:

Samples:

The instrument provides calculated sample results in mg/L, calculations are only necessary if a dilution was used.

Raw Data Value (mg/L) x Dilution Factor = Total Cyanide (mg/L)

Accuracy:

A matrix spike and matrix spike duplicate must be performed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for accuracy.

Spike calculation:

$$\% \text{ Recovery} = \frac{SSR-SR}{SA}$$

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

Precision:

A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for precision.

Use relative percent difference (RPD) calculation:

$$RPD = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

MS = Matrix Spike Value
MSD = Matrix Spike Duplicate Value

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

Standard Operating Procedure

TITLE: Thiocyanate Determination

DEPARTMENT: Inorganic-Wet Chemistry

APPLICATION: Surface, groundwaters and domestic and industrial wastewaters with concentrations from 0.1 to 2.0 mg SCN^-/L.

REFERENCE: SM4500 CN-M, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992

PROCEDURE SUMMARY:

The water is acidified to pH 2 by adding concentrated HNO_3 and is passed through an adsorption column, if pretreatment is needed. A Ferric Nitrate solution is added to the eluate, which in the presence of Thiocyanate, produces an intense red color. The concentration of Thiocyanate can be determined by reading the absorbance of the sample vs. a standard curve at 460 nm on the spectrophotometer.

REPORTING LIMIT: 0.1 mg SCN^-/L.

REVIEWED BY: Jeffrey A. Gordon — Date: 10-11-00
Jeffrey A Gordon
Inorganic Section Supervisor

Gregory J. Graf — Date: 10-13-00
Gregory J. Graf
Quality Control Officer

APPROVED BY: Glen A. Coder — Date: 10-13-2000
Glen A. Coder
Laboratory Manager

I. SAMPLE HANDLING AND PRESERVATION:

Collect sample in a plastic or glass container, and preserve samples at pH <2 with nitric acid and refrigerate to 4 °C. The method cited lists no holding time. A holding time of 14 days will be used

II. INTERFERENCES:

Hexavalent Chromium: Eliminate Hexavalent Chromium by adding Ferrous Sulfate and raising the pH to 9 with 1N Sodium Hydroxide, which precipitates Fe^{3+} and Cr^{3+}. Filter the sample.

Reducing agents that reduce Fe^{3+} and Fe^{2+}: Add 3-4 drops of Hydrogen Peroxide Avoid excess Hydrogen Peroxide to prevent a reaction with SCN^-.

Industrial Wastes that are highly colored or contain various organic compounds may contain interferences. Pretreat the sample to eliminate the interferences

III. APPARATUS AND MATERIALS:

Hach Spectrophotometer DR 2000
50mL buret
Glass wool
Powder funnel
Plastic tubing

IV. REAGENTS:

Ferric Nitrate Solution: Dissolve 404 g $Fe(NO_3)_3$ $9H_2O$ in 800 mL D.I. water. Add 80 mL concentrated HNO_3 and dilute to 1 L. Shelf life is one year.

Nitric Acid Solution, 0 1N· Mix 6.4 mL concentrated HNO_3 in 800 mL D.I. water Shelf life is one year.

Stock Thiocyanate Solution: Dissolve 1.673 g Potassium Thiocyanate (KSCN) in D.I. water and dilute to 1000 mL. 1.00 mL = 1.00 mg SCN^-. Shelf life is one year

Standard Thiocyanate Solution: Dilute 10 mL stock solution to 1 L with D.I. water; 1.00mL = 0.01 mg SCN^-.

Sodium Hydroxide Solution, 4 g/L: Dissolve 4 g NaOH in 800 mL D.I. water and dilute to 1 L. Shelf life is one year.

Macroreticular Resin, 18 to 50 mesh (Amberlite DAX-8): The resin must be purified prior to use.

Methanol, Acetone and Hexane, pesticide grade.

Purifying Resin:

Place enough resin to fill the column into a beaker and add five times the resin volume of acetone. Stir gently for one hour and let settle. Pour off particulates and acetone

Add five times the resin volume of hexane and stir for one hour. Pour off particulates and hexane, and add five times the resin volume of methanol. Stir for fifteen minutes and then pour off. Add three times the resin volume of 0.1N NaOH solution, and stir for fifteen minutes.

Add three times the resin volume of 0.1N HNO_3 solution, and stir for fifteen minutes. Pour off the HNO_3 solution, and add three times the resin volume of D.I. water. Stir for fifteen minutes. Drain the extra water and fill the column with purified resin Store excess purified resin, after covering it with D.I. water, in a closed jar

Prepare Working Thyiocyanate Standards:

Pipette the indicated amounts of standard 10 mg/L KSCN solution into 200 mL volumetric flask and dilute to volume with D.I. water. Prepare fresh at the time of calibration. The Hach Spectrophotometer can store the calibration curve The curve shall be renewed quarterly or as required.

mL Standard Solution	SCN^-, mg/L
0.0	Blank
0.50	0.10
1.25	0.25
2.50	0.50
5.0	1.00
10.0	2.00

Standards for LCS and ICV/CCV:

ICV - Initial Calibration Verification
CCV - Continuing Calibration Verification
LCS - Laboratory Control Sample

ICV/CCV: 1.00 mg/L
LCS: 0.50 mg/L

Preparation of the Matrix Spike and Matrix Spike Duplicate: 0.50 mg/L

V. SAMPLE PRETREATMENT:

Acidify 150 mL of sample to pH 2 by adding concentrated HNO3 dropwise while stirring.

Measure 90 mL of sample in a graduated cylinder. From this add and drain five separate 5 mL volumes of solution and drain to the approximate bed height. Sample should pass through the column at a flow rate of 20 mL/minute or less. Discard eluate Then pour the remainder of the 90 mL into the column. Add the 60 mL remaining from the sample and let 60 mL of eluate pass through the column. Collect the next 60 mL to be tested.

When resin becomes packed and the flow rate falls to 4-5 mL/minute, use gentle pressure through a hand pump or squeeze bulb to the column. Use a separator funnel for the liquid reservoir. Do not let the liquid level drop below the absorbent in the column.

Everyday the column is used, a mid-range standard should be prepared to check the absorption curve.

Regenerate the column between samples by rinsing with 100 mL 0.1 NaOH; 50 mL 0 1 HNO_3 and 100 mL DI water. Rinse the empty glass section of the buret with the water. For complete regeneration rinse with 100 mL methanol. Leave column covered with last rinse water for storage.

VI. PROCEDURE:

1.) Use filtered sample or a portion of a diluted sample to insure the concentration of SCN^- is between 0 1 and 2 mg/L. Adjust the pH to 2 with concentrated HNO_3 dropwise, if not already preserved.

2) Fill a 25 mL absorption cell and measure absorbance at 460 nm against a reagent blank. Subtract this value from the reading with the Ferric nitrate reagent to eliminate any absorbance from colored samples

3.) Pipet 50 mL into a beaker and add 2 5 mL Ferric nitrate, and mix well. Measure the absorbance of the developed color within five minutes from adding the reagent. The color develops within 30 seconds, and will fade when exposed to light.

VII. CALIBRATION:

The linear range for the method is from 0.0 to 2.0 mg/L. An established curve with a correlation coefficient of 0.995 or better will be stored in the memory of the spectrophotometer. At the beginning of each run an ICV and LCS will confirm the validity of the calibration. If either fall outside the QC limits, a new calibration curve will be established.

VIII QUALITY CONTROL:

Initial Calibration Verification (ICV):
The ICV must be run immediately after calibration and meet 90-110% control limits If not, recalibrate

Initial Calibration Blank (ICB):
The ICB must be analyzed after the ICV and be less than the absolute value of the estimated quantitation limit (EQL), 0.1 mg/L. If not, recalibrate.

Laboratory Control Sample (LCS):
A standard must be analyzed for each 20 samples, and meet control limits** (80 - 120%).

Method Blank:
The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <EQL, 0.1 mg/L. Other criteria may apply, such a regulatory limit and analyte concentration in samples.

Continuing Calibration Verification (CCV):
The CCV is analyzed after every ten analytical samples and meet 90-110% control limits. If it is not within control, stop analysis, and recalibrate. All samples analyzed since the last successful CCV must be reanalyzed.

Continuing Calibration Blank (CCB):
The CCB is analyzed after every CCV and must be less than the absolute value of the EQL. If it is not within control, stop analysis, and recalibrate All samples analyzed since the last successful CCB must be reanalyzed.

Matrix Spike:
A spike must be performed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the control limits** of (75 - 125%).

Matrix Spike Duplicate:
A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits for accuracy and the Relative Percent Difference (RPD) between the MS and MSD must meet the control limit** of 20% or less.

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to data validators.

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE: Total Recoverable Phenolics, Automated Analysis

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION. Distilled samples of waters, wastewaters, leachates and soils

REFERENCES: EPA 600 4-79-020, March 1979, Method 420.2
EPA Manual SW-846, 3rd Edition, Method 9066

PROCEDURE SUMMARY:

Upon distillation of the sample, volatile phenols are removed from their original matrix and collected The distillate is analyzed. Phenol, ortho- and meta-substituted phenols, and para-substituted phenols where the para- group is carboxyl, a halogen, a methoxyl, or a sulfuric acid group are all determined by reaction with 4-aminoantipyrine. Not determined are para-cresol, and other para-substituted phenols where the para- group is an alkyl, an aryl, a nitro, a benzoyl, a nitroso, or an aldehyde group. The method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide and 4-aminoantipyrine to form a red complex which is measured at 500 nm.

REPORTING LIMITS· Water: 0.010 mg/L; Soil 0.5 mg/kg

REVIEWED BY: Jeffrey A. Gordon — Date: 10-11-00
Jeffrey A Gordon
Inorganic Section Supervisor

Gregory J. Graf — Date: 10-13-00
Gregory J. Graf
Quality Control Coordinator

APPROVED BY: Glen A. Coder — Date: 10-13-2000
Glen A. Coder
Laboratory Manager

SAMPLE HANDLING AND PRESERVATION.

Samples are collected in glass bottles and preserved with sulfuric acid to a pH of <2 Holding time is 28 days from the date of collection. After distillation, samples should be capped, kept at 4 °C, and analyzed within five days.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

INTERFERENCES:

Interferences are eliminated or reduced to a minimum upon distillation. Color response of phenolic materials with 4-aminoantipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason, phenol (C_6H_5OH) has been selected as a standard and any color produced by other phenolic compounds is reported as phenol. Because this substitution generally reduces response, this value represents the minimum concentration of phenolic compounds.

To avoid background contamination of leachable phenolics from plastic:

- Transmission tubing should be replaced with Teflon manifold tubing (0.8 mm inner diameter).
- Regular plastic pump tubing should be replaced with duraprene pump tubing: green for carrier, and orange-white for color reagent and buffer.
- Use glass containers for reagents, unless, upon verification it is found that a particular container does not contain leachable phenolics, then this container may be used.
- If the blank peak is negative, the carrier contains a greater concentration of phenolics than the blank. If the blank peak is positive, the blank contains a greater concentration than the carrier.

APPARATUS AND MATERIALS:

Analytical balance capable of accurately weighing to the nearest 0.0001 gram
LACHAT Quikchem Automated Ion Analyzer
500 nm interference filter
150 cm sample loop
10-210-00-1-A phenol manifold
Volumetric flasks: 100 mL, 200 mL, 250 mL, 1000 mL
Pipettors: adjustable and fixed volume

REAGENTS:

Deionized (D.I.) water
4-aminoantipyrine [$CH_3NN(C_6H_5)COC(HN_2):CCH_3$]
Potassium ferricyanide ($K_3Fe(CN)_6$
Potassium chloride (KCL)
Boric acid
Sodium hydroxide (NaOH)
Phenol
Copper sulfate
Phosphoric acid
Helium gas
APG reference solution

Prepare 4-AAP Solution

. Dissolve 0.1625 grams 4-aminoantipyrine in approximately 200 mL D.I water and dilute to 250 mL. Prepare fresh daily.

Prepare 1M Sodium Hydroxide, NaOH

. Dissolve 40 grams NaOH in 800 mL D.I. water in a liter flask Dilute to volume. Shelf Life = 1 year.

Prepare Buffered Potassium Ferricyanide, pH 10.3

. In a 1 liter volumetric flask, dissolve 2.0 grams potassium ferricyanide, [$K_3Fe(CNO_6$], 3.1 grams boric acid (H_3BO_3) and 3.75 grams potassium chloride (KCl) in about 800 mL D I. water. Add 47 mL 1M sodium hydroxide. Dilute to 1 liter. This solution is good for two to three months.

Carrier is helium degassed D.I. water

Prepare Stock Phenol Standard, 1000 mg/L

. Dissolve 1.00 gram phenol in 500 mL D.I. water.

. Add 1 gram copper sulfate and 0.5 mL concentrated phosphoric acid as a preservative Dilute to 1 Liter.

. Prepare two from two different sources. Shelf life = 1 year.

Prepare Working Standards, 10 mg/L

Dilute 10 mL stock phenol solution to 1 liter Shelf life = 1 week

Prepare Calibration Standards

Into four 100 mL volumetric flasks, pipette 2 0, 1.0, 0.5 and 0.10 mL of 10 mg/L working standard solution. Dilute to the mark with D.I. water. This makes standards of 0.20, 0.10, 0.050 and 0.010 mg phenol/L respectively. Shelf life = 1 day.

Prepare ICV/CCV Standard

. Into one 100 mL volumetric flask, pipette 1.0 mL of 10 mg/L working standard from alternate source Dilute to mark with D I water Shelf life = 1 day

PROCEDURE.

1. Set up instrument (See En Chem Method, WCM-29)
2. Connect 10-2100-00-1-A phenol manifold
3. Install 500 nm interference filter.
4. Install 150 cm loop.
5. Place proper waste line into container for collecting cyanide waste from phenol analysis

   Samples which exceed the linear calibration range must be diluted.

QUALITY CONTROL:

Calibration/Linear Range
The instrument must be calibrated every time it is set up. Calibration standards are (mg/L): 0 010, 0.050, 0.10 and 0.20.

Correlation Coefficient (r-value)
The correlation coefficient, the measure of linearity of a standard curve, must be 0.995 or greater. If the value is less than 0.995 recalibrate the instrument.

Initial Calibration Verification (ICV)
The ICV must be analyzed immediately after calibration and meet the rejection criteria of $\pm$ 10% of the true value. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be $\leq$EQL. Recalibrate if it fails.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples Rejection criteria is ± 10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be ≤EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

Laboratory Control Sample (LCS)
The LCS (APG reference solution) is carried through all preparation procedures and analyzed for each matrix type with a frequency of 5% In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be reprepared and reanalyzed. The current control limits** are: Water 61-143%; Soil 80-120%.

Method Blank (MB)
A MB is carried through all prep procedures and analyzed with a frequency of 5% Rejection criteria is <LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

ACCURACY

One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. The current control limits** are: Water 68-128; Soil 68-133.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with an [N qualifier. The control limits for a post-spike are 75-125%.

If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained. If only the matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding parent sample is flagged with an [MS qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however, an [N qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and LCS DUP must be used in its place.

PRECISION

Matrix spike duplicate samples are analyzed 1 per batch or at a frequency of 5%, for samples that are similar in matrix.

For matrix spike duplicate samples, relative percent difference (RPD) is used to calculate compliance. The current control limits** are: Water 0-17; Soil 0-22.

Calculation:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with an [* flag

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to data validators.

Sample Result Calculations:

Aqueous Sample Calculation:

Raw Data result (mg/L) X DF = Final Result (mg/L)

Soil Sample Calculation:

$$\frac{\text{Raw Data result } (\mu g/L) \times FV\,(L) \times DF}{\text{sample weight (g)} \times \text{dry wt.(decimal form)}} = \text{Final Result (mg/kg dry weight corrected)}$$

DF = Dilution Factor
FV = Final Volume

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

En Chem, Inc.

SET No: 1

Quality Assurance Document

INSTRUMENT OPERATING PROCEDURE

INSTRUMENT TYPE: Automated Ion Analyzer System

MANUFACTURER: Lachat

MODEL: QuikChem 8000

SERIAL NO.: 950420-01

DEPARTMENT: Inorganics - Wet Chemistry

PROCEDURE:

1. Turn on computer with ON/OFF button. Turn on printer. The power to the monitor, autosampler and reaction module is controlled with a multi-outlet noise filter and surge suppresser located behind the reaction module. Turn the red switch to "ON".

2. Allow the autosampler to perform its startup routine and wait until the autosampler stops with the probe above the wash bath.

3. Log into the Network with your Login name and password. Windows program will be activated. Program Manager screen will appear. Click twice on Lachat Instruments. On the Lachat Instruments window, click twice on the Omnion FIA icon. The startup box will come up, then click on **'OK'**. The autosampler probe will go into the wash bath and dilutor activity may be heard. The injection valves may turn to the Inject state if they were not already there.

4. To encourage GALP (Good Automated Laboratory Practice), Omnion requires that the operator "Log-in" every time with name and password. Enter your assigned name and password in the correct case in the spaces indicated and then click on the **'OK'** button.

5. After you have logged in, the Omnion FIA Main Menu will appear on the screen. The header should contain the serial number of the software key. No messages should be seen about the SS420 boards, or any serial port failures. Click on the Instrument button labeled 'FIA Instrument 1'.

6. Each valve allocated to this instrument will be cycled from Load to Inject and Load to Inject again. This is a test of the valves and if no messages are received about failing valves, then they are OK.

7. If a valve fails, a box will tell you which channel's valve failed. You can press OK and continue. You should arrange for service for the failing valve by calling Lachat Instruments.

8. After the injection valves are tested, an instrument window for FIA Instrument 1 will appear. Omnion will automatically open the last Method, Tray, DQM Plan and Data File used. Arrow up to 'File" and click. When window appears click on **'Open Method'**.

   Under Open Method File Directories, click on **c:\omnion\methods** and then click on the method you will be running. A list of methods will appear. Highlight the appropriate one in the **File Name** box and click on **'OK'**.

9. The Method will open and the **Analyte Table** will appear. It will show the Analyte Name and which Channel it is run on. The concentration Units and concentration of the calibration standards will be listed. To the far right will be the timing periods which the instrument uses to detect the peaks.

10. Click on **'File'** again and then **'Open Tray'**. The **'Open Tray File'** window will come up. Directories will be **c:\omnion\trays\method**. Click on the method you will be running. A list of previous trays for that particular method will appear. Click on a recent **tray file name** and the on **'OK'** to call it up. Edit this tray below the Calibration Standards with the sample numbers you will be running. Also edit any dilutions that will be required.

11. Click on **Save Tray As** and edit name of tray to reflect toady's' date and letter to reflect which run: YYMMDDL. L is the sequence of the tray that day i.e., a=first tray, b=second tray and so on. Example: 960730a.

12. Click on **File** again and then on **Open DQM**. The Open DQM File window will come up. Directories will be **c:\omnion\DQM\method**. Click on the method you will be running and then on the method **DQM File Name**. Click on **OK** and the DQM (Data Quality Management) Plan will appear.

    This DQM Plan supplements the tray table itself and acts as a standard operating procedure to be used when running this tray. Check the DQM to make sure it will apply to this tray, or edit it if necessary. If any changes were necessary then the DQM should be saved, and then replace the DQM in the tray, to make the changes permanent. If no changes were necessary, then minimize this window.

13. Click on the **Run Tray** button. The Tray Run Window will appear. Leave the **Method** and **Tray** spaces blank to run the currently loaded Method and tray. You must fill in the **Data File** space so that Omnion knows where to put the data it will generate. Click on **Catalog** after the **Data File** space to see a list of previous data files. Click on an old file name and then edit it to match today's tray name. Click on **OK**.

14. Insert the proper manifold for the desired analysis to the proper channel specified in the Analyte Table. The load/inject valve for each channel has six connections with the following designations:

Port #	Function
1	Sample Loop Inlet

Port #	Function (continued)
2	Carrier Stream Inlet (From Pump) (Short section of plastic tubing remains port #2 at all times)
3	Outlet to reaction manifold
4	Sample loop outlet
5	Sample loop waste line
6	Sample source line (from sampler, pump)

Each reaction manifold has a line with pump tubing attached labeled "carrier". Disconnect the pump tubing from this line and reconnect it to the section of line that comes from port #2. Connect the section of tubing coming from the reaction manifold to port #3.

15. Connect the outlet line from the manifold, located top right, to the bottom inlet of the flow cell.

16. Insert backpressure loop if required, between top outlet of flow cell and the waste line.

17. Place waste line into proper waste collection container.

18. Install proper interference filter.

19. If required, allow heating unit to heat to correct temperature.

20. Install a proper length sample loop in ports #1 and # 4.

21. Install the pump tubing onto pump cartridges. Check for wear on the pump tubing by rolling between thumb and fingers when stretched across a pump cartridge. Replace tubing if "flat" spots are felt.

22. Move tension levers to left to provide proper tension.

23. Check all connections for leakage and proper flow. Make sure waste lines are routed to proper container.

24. Fill standard, check standard and blank vials with proper solutions. Fill sample tubes with proper samples.

25. When ready, click on **RUN** on the Tray Run Window and the analysis will start. The symbol for the tray on the toolbar will turn to a **STOP** sign until analysis is complete.

26. If necessary to stop the run anytime, click on the **STOP** sign. A window will come down with the options to <u>RESUME</u> or <u>ABORT TRAY.</u>

27. After instrument has calibrated, click on the **Review Calibration** button on the toolbar. The graph of the calibration will be displayed along with the area of each standard. If the r value of the calibration is 0.995 or better, print the calibration graph and allow the analysis to continue.

28. After analysis is completed the **STOP** sign in the Toolbar will be replaced with **Run Tray.** Click on File and the window will come down. Click on Export Data. Export data window will appear. Highlight whatever Data Items and Channel Dependent Data Items you want exported to EXCEL Save Export Data As window will appear. Directories should be g:\data\inorgani\lachat\archives.

    Highlight the .text file you have just run in the File Name box. Click on **OK** and data will be exported to EXCEL. Then go back and click on File again. When window comes down, click on Open Runtime Rpt. Open Runtime Report File window will appear. It should have current data, method and file name of analysis just completed, or if going back at a later date, then highlight the one that you want. Click on **OK** and the Runtime Report will appear on the screen. Click on File and then on Print/Export Runtime Rpt. Runtime Report window will appear.

    Click on **OK** to print. On Print window choose the Print Range you want. **ALL** is normally highlighted. Click on **OK**.

29. After analysis is complete, place reagent lines in D.I. water and rinse several minutes. Remove lines from water and pump air through the lines until dry.

30. Turn off pump, release tension levers on pump cartridges and remove pump tubing from cartridges.

31. Disconnect pump tubing to port #2 at the pump tubing connector, disconnect line to port #3 at port #3 connector and connect pump tubing from #2 to line from manifold.

32. Disconnect manifold outlet line from flow cell inlet connector.

33. Wrap pump tubing lightly around manifold panel and store in appropriate box, with sample loop and interference filter.

NOTE: Any daily or periodic maintenance must be recorded in the instrument daily log book.

SHUTDOWN:

1. Click on **EXIT** icon

2. Omnion FIA Data System Window appears. Click on **Logoff.**

2. Omnion FIA Data System Window appears. Click on **Logoff.**

3. Window listing User Name and question: **Logoff Current Session?** Click on **YES**.

4. Window for User Name and Password comes up. Click on **EXIT**.

5. Window comes up with: **Terminate Data System?** Click on **YES.**

6. Turn off computer, printer and switch on surge suppresser.

REVIEWED BY: Jeffrey A. Gordon
Jeffrey A. Gordon
Inorganic Section Supervisor

3-24-00
Date

Gregory J. Graf
Gregory J. Graf
Quality Assurance Officer

3-28-00
Date

APPROVED BY: Glen A. Coder
Glen A. Coder
Laboratory Manager

3-29-00
Date

En Chem, Inc.

Quality Assurance Document

ANALYTICAL METHOD

TITLE: Total Suspended Solids Dried at 103-105 °C
Total Volatile Suspended Solids Dried at 550 °C

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Ground Water, Surface, Drinking and Saline Waters; Domestic and Industrial Wastewaters

REFERENCES: EPA 600 4-79-020, Revised March 1983, Method 160 2 (TSS) and 160.4 (TVSS)

PROCEDURE SUMMARY:

A well-mixed sample is filtered through a glass fiber filter and the residue retained on the filter is dried to a constant weight at 103-105 °C. Estimated Quantitation Limit (EQL) for this analysis is 10 mg/L.

If Total Volatile Suspended Solids are requested:
The residue from the Total Suspended Solids is ignited to constant weight at 550 °C $\pm$ 50 °C. The weight lost on ignition is the volatile suspended solid. Estimated Quantitation Limit (EQL) for this analysis is 10 mg/L.

REVIEWED BY: Jeffrey A. Gordon, Inorganic Section Supervisor — Date: 10-11-00

Gregory J. Graf, Quality Assurance Officer — Date: 10-13-00

APPROVED BY: Glen A. Coder, Laboratory Manager — Date: 10-13-2000

SAMPLE HANDLING AND PRESERVATION:

Samples should be stored at 4 °C until analysis Warm to 20 °C before analysis. Hold time for samples is 7 days from time of collection. Plastic or glass containers are adequate.

APPARATUS AND MATERIALS:

Filter holder: Gelman 35 mm filtration diameter
Vacuum source Marathon pump 1/3 HP, 1725 rpm
Filter: Whatman 934-AH glass microfibre, 4.25 diameter (PreWeighed)
Manifold: Gelman 4205
Filtering flask
Forceps, flat blade
Crucible tongs
Analytical Balance: Mettler AE 160, 0.1 mg capability
Drying oven
Muffle furnace
Aluminum evaporating dishes for filters
Desiccator
Indicating Drierite
APG reference solution

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

PROCEDURE:

1. Preparation of Glass-fiber Filter Disk

   a. Place PreWeighed filter on filtering apparatus with wrinkled side up Record weight of filter on the electronic gravimetric bench sheet.

   b. Apply vacuum and wash three times with 20 mL deionized (D I.) water.

   c. Continue the vacuum to ensure all traces of water have passed through the filter.

2. Sample Analysis

   a. Use a graduated cylinder and measure out 100 mL of a well mixed sample.

Record volume on the electronic gravimetric bench sheet

NOTE: Volume to filter - Use a volume less than 100 mL if it appears that plugging of the filter may occur. A weighable quantity of 50 -100 mg is a desirable range to obtain without plugging.

b. Draw the volume through the filter with vacuum. Wash three times with 10-15 mL aliquots of D.I. water, taking care to rinse solids from the cylinder and the filter apparatus sides onto the filter.

c. Release vacuum. Place filter in the labeled aluminum dish. Put in drying oven at 103-105 °C for a MINIMUM of 2 hours to insure constant weight.

d. Cool in desiccator and weigh the filter. Record final weight on the electronic gravimetric benchsheet. Consecutive final weighings must be conducted until a weight change between final weighings of < 0.5 mg is obtained. Wait at least 60 minutes between final weighings. Place filter back in the aluminum dish and store in the dessicator between weighings

If volatile suspended solids are to be analyzed

a Place aluminum dish and filter in muffle furnace at 550°C for 15 minutes.

b. Cool in desiccator and weigh filter as before. Record final weight on the electronic gravimetric benchsheet. Consecutive final weighings must be conducted until a weight change between final weighings of < 0.5 mg is obtained. Wait at least 30 minutes between final weighings. Place filter back in the aluminum dish and store in the dessicator between weighings.

Calculations

Total Suspended Solids

Example:

Volume, (mL)	100
Final Weight	1.4275
-Initial Weight	1.4180
Suspended Solids, SS	0.0095

$$\text{TSS, mg/L} = \frac{SS \times 10^6}{\text{Volume, mL}} = 95$$

Volatile Suspended Solids

Final Weight	1.4275
-Ignited Weight	1.4188
Volatile Solids, VSS	0 0087

$$\text{mg/L} = \frac{\text{VSS} \times 10^6}{\text{Volume, mL}} = 87$$

$$\text{\% Volatile} = \frac{\text{VSS (mg/L)} \times 100}{\text{SS (mg/L)}} = 92$$

QUALITY CONTROL:

Laboratory Control Sample (LCS)
An LCS reference consisting of a known result must be prepared and analyzed for each matrix type and meet the current control limits This material should be from APG or other external vendors. Rejection Criteria. If the LCS does not meet current control limits, terminate analysis The current control limits** are 74-114%.

Precision
A duplicate must be analyzed on each group of samples of a similar matrix type at a frequency of 5%, or 1 in 20 samples. The current control limit** (RPD) is 0-23%.

Duplicate calculations:

If the sample value, the duplicate value, or both are less than 5 times the EQL, use the absolute difference (AD).

| Sample - Duplicate | = AD

If both the sample value and the duplicate value are equal to or greater than 5 times the EQL, use the relative percent difference (RPD).

$$\text{RPD} = \frac{S - D}{(S+D)/2}$$

S Original sample value
D Duplicate sample value

Method Blank (MB)
A MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

**Control limits are updated periodically. The control limits that are in use at the time of analysis will used and made available to data validators.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

PENETROMETER TEST PROCEDURES

The following procedures are used by STRATIGRAPHICS in performing penetrometer testing.

1. The sounding location is evaluated for surface obstructions or overhead utilities. The local traffic situation is also evaluated. The Client Representative is queried if he has obtained all subsurface utility clearances in area. Client's authorization to proceed with subsurface exploration at location is received.

2. The penetrometer rig is moved onto location. A laser alignment beam is used to position the rig on location if an ultra-high (less than 0.5 inch) degree of accuracy is required, such as during CPT through cored holes in pavement. For most work, visual alignment is used for positioning, typically placing the rig within 1 ft of the planned location. Crew notes down in field log sheets any unusual setup situations, including pavement or cored hole.

3. The zone immediately around the test point is checked for proper seating of grout seals for use in pressure grouting the hole. Surface leveling is performed if a seal might not be developed. Dry granular grout is used to develop a seal on a rough surface that cannot be leveled.

4. The penetrometer rig is leveled using the hydraulic leveling system. Rig lateral and longitudinal levels are checked using a bubble or electronic level placed on the rod clamp. Once rig is leveled, begin GPS data logging, if required. Check lightning detector antenna alignment, if it is being used.

5. The CPTU-EC penetrometer is visually observed for wear or damage. If a piezometric sounding is to be performed, the piezometer elements are de-aired using the vacuum saturation system. Piezometer filters are replaced before every sounding if clays were encountered in the previous sounding or if filter damage or clogging is observed. A maximum of three soundings can be performed with a single filter set if filter damage or clogging is not observed.

    a) The CPTU-EC piezometric filters are de-aired using the vacuum saturation system until few bubbles are observed flowing from the filters. Piezometric transducer output is checked versus vacuum gauge pressure. In general, use of the hi-vacuum electric pump results in -0.98 to -1.00 tsf of vacuum measured at the transducer. A vacuum of -0.92 to -0.96 tsf is typically measured when de-airing filters using the air operated vacuum pump.

    b) piezometric response is checked by developing a full vacuum in the saturation chamber, followed by quick release of vacuum to atmospheric pressure. Transducer response should reflect this change within about 2 seconds. Much slower response indicates poor saturation or a clogged filter. Continue saturation or change filters. Crew notes down on field log sheets whether piezometer saturation procedures were performed.

6. The soil electrical conductivity (EC) module is checked by applying a series of calibration resistors across the electrodes and monitoring EC channel output. High output (+15,000 uS/cm) in air indicates an electrode short to case. Low output or no output when the resistor is applied to electrodes indicates an open circuit. Crew notes on field log sheets which calibration resistor was used and what reading was obtained during the EC check.

7. The UVF module consists of a sapphire window in the side of the penetrometer, a UV light source, filters, and photonic sensors. The UV light source illuminates the soil next to the window. If the soil contains compounds, such as petroleum hydrocarbons, that fluoresce, the resulting light can be detected with photonic sensors. The intensity of the fluorescence can often be related to the concentration of contamination in situ.

   The UV light source is bandpass filtered at 254 nm to provide a light of very narrow wavelength for excitation of soil compounds next to the sapphire window. The photonic sensor is high pass filtered at 290 nm to sense resulting flourescence from soil compounds, next to the sapphire window, under the excitation of the 254 nm UV light source.

   Prior to performance of a CPT-EC-UVF sounding, the operator turns on the UV light source, and allows at least 5 minutes of warmup time, before performing subsurface exploration. Other calibration procedures may be performed during this warmup period.

   The photonic sensor operation is checked by exposing the sapphire window to ambient light within the penetrometer rig. A high light signal of about 4.500 Volts is obtained under ambient light conditions. A light blocking magnet is then used to cover the sapphire window, cutting off ambient light to the photonics sensor. A dark signal of about 0.200 to 0.500 Volts is obtained with the blocking magnet covering the window. The blocking magnet is then turned over and applied to the window. A flourescent target, mounted on the back of the magnet, is thus exposed to the UV light, and results in a high target sensor output of about 3.000 to 4.000 Volts. If the UV light source is not operational, only a dark signal level will be obtained during this step.

   Finally, the CPT-EC-UVF penetrometer is lowered into the rig centerwell as part of beginning subsurface exploration. While the penetrometer and UVF window are inside the centerwell, and the centering guides have been mounted around the rod string (see Step 9), the operator checks the zero output of the UVF sensor. Since the steel centerwell is opaque to light, and sealed at the bottom with rubber seals, and blocked at the top with steel centering guides, the centerwell provides a repeatible, very low light level environment for zeroing the UVF sensor. If sensor output is greater than +/- 0.100 Volts, the operator should reset zero levels for the sensor. Once the UVF window is below the ground surface, sensor output may be below zero levels (-0.100 to -0.300 Volts) as light levels in the soil may be less than in the centerwell. However, the centerwell is used for zeroing, as it provides a repeatible very low level light condition.

8. The penetrometer load cells are checked by manually loading the penetrometer and noting a positive response of about 10 to 50 lbs on both cone tip and friction (total) load cell outputs. The friction loadcell should be checked next by manually pushing on it while avoiding the cone tip. Total loadcell output should change, with little change in cone tip loadcell output.

   The shunt calibration is used at this point to verify proper transducer response. When pushing the shunt cal button, a full scale response of about 23,000 lbs should register on both load cell outputs for the S1510 series penetrometers. A full scale output of about 26,000 lbs should register on both load cell outputs for the S1500 series penetrometers. A full scale output on channels Cone1 and Total1 of 32,768 lbs is registered for S2000 series penetrometers, with about 59,000 and 75,000 lbs registering on Cone2 and Total2, repectively.

   Inconsistent and floating output can be indicative of damage to transducers, especially caused by water flooding of the penetrometer. This gets worse with time. Penetrometers must be changed immediately in this situation.

Changes in loadcell output when the penetrometer is grounded to the rig indicate ground loop problems, possibly caused by moisture in the penetrometer. If small changes occur, the penetrometer might be temporarily kept in operation, with close attention paid to zero load consistency. Output of full scale readings, typically +/- 22,000 or +/- 26,000 lbs indicate an open circuit. Check connectors for fit and cabling for cuts in this situation.

9. Check depth encoder operation by rotating encoder wheels. Make sure that encoder temperature is greater than 32 degree F. The encoder may not work consistently if it is colder than this. Check encoder wheels to make sure they are free of all oils, and are relatively clean and dry. If wheels are oily, clean wheels with alcohol wipes, making sure wheels are absolutely dry after cleaning.

   The penetrometer is clamped into the push system at this time and the first two sections of sounding rod are added. The penetrometer is then lowered into the centerwell. Centering guides are fitted around the sounding rods when the enlarged portion of the penetrometer has passed the top of the centerwell. Centering rings are fitted to the rod string after the centering guides have been lowered below the top of the centerwell. (See section on UVF zeroing).

   Lowering of the penetrometer to the grout seal is monitored using the closed circuit camera and monitor (CCTV). The penetrometer is lowered until the conical tip is level with the grout seal and the depth encoder is reset. The rod string stickup, relative to the push table, is measured at this point.

   The penetrometer is raised about 4 inches at this point, to ensure that no loads are being applied to the loadcells during autozero procedures. Crew notes in field log sheets all transducer start zero readings, including penetrometer temperature, if the penetrometer includes a thermal sensor. Data logging software is initialized by entering sounding file name.

10. Data logging software is initiated by keystroke command. The software autozeros the cone and friction (total) loadcells. The operator counts 5 seconds, pushes the shunt cal button for another 5 seconds, and begins the sounding. This procedure permanently records initial zeros and shunt calibration values onto the sounding disk file. Operator verifies that loadcell values of about 33,000 (S2000 series), 23,000 (S1510 series) or 26,000 (S1500 series) lbs are displayed during the shunt cal procedure.

11. Begin pushing the penetrometer into the ground. Observe, using the CCTV, the verticality of the initial penetrometer push. Do not continue sounding if significant (+1/2 inch) deviation from vertical occurs. Begin pumping grout, if required, after penetrometer is about 3 to 5 ft in ground. Do not allow grout to get on piezometric elements as the grout will clog filters.

12. Continue adding rods and pushing penetrometer into the ground while monitoring hydraulic pressures and transducer on-screen display. Observe loadcell output during rodbreaks (while rod clamp is released to add next rod). Friction output can go negative, but cone end bearing should never go negative, other than during rod string pullback. Note down depths at which negative cone tip output occurs. Pump in about 2 pump strokes of grout for every 5 to 8 rods added to string.

   Monitor that depth encoder is performing properly by noting changes in depth. Depth change at 1 Hz logging frequency should be about 0.08 ft per scan. Depth change per sounding rod added is 3.28 feet. If much less depth change is occurring, stop and inspect encoder wheels. Oil on wheels can cause slippage. Clean wheels with alcohol and dry extremely thoroughly. Use hot air drier if possible. If slippage has occurred, note down all rod break depths for rest of sounding, or until all signs of slippage have stopped. Repeat wheel cleaning if slippage re-occurs.

13. At target depth or refusal, count number of rods left in rack and measure stickup relative to push table and record values. Compute actual depth of sounding and note on field log sheet. End data logging and begin processing log.

14. If rod string decontamination is to be performed, make sure steam cleaner feed pump is on and steam cleaner is set to auto mode. Begin pulling rods. Monitor waste water container. Grout hole using about 2 pump strokes every 3 rods pulled.

15. Pull penetrometer to about 3 inches above grout seal. Note down all transducer end zeros and penetrometer temperature. Lower penetrometer into grout seal to plug it. Pump in additional grout until hole is full. Note total amount of grout that was pumped into the hole. About 1/2 to 3/4 of a gallon are needed to grout 10 ft of hole.

16. Leave steam cleaner on as penetrometer is pulled through rod washer if piezometer saturation is to be performed, or if penetrometer is to be handled. Turn off steam cleaner and rinse penetrometer with cold water from pressure hose if a sounding not requiring piezometer saturation is to be performed immediately.

17. Finish processing sounding log. Shut down GPS logging, if performed. Strap rod rack. Lower truck back onto ground and pull away from hole. Check grout level. Add dry granular grout to make up for liquid grout penetration into permeable strata. Note down how much grout was added. Patch pavement, if required. Clean area, if required.

18. Repeat steps 1 to 17 for next sounding.

QUALITY ASSURANCE

The key to quality assurance is system design, operator experience and adherence to procedures. STRATIGRAPHICS penetrometers are simple, robust tools, including features to maximize depth capability while assuring high data quality. Data logging equipment features ultra high 16 bit A/D resolution. Andrew Strutynsky, system designer and operator, has years of experience in performing penetrometer testing.

Test data are monitored by the engineer as the CPTU-EC-UVF soundings are performed. Field results may be used immediately to guide concurrent drilling and sampling operations. Data are recorded on floppy and hard disk. Measured channels consist of: depth, time, cone end bearing resistance, friction sleeve resistance, piezometric pressure, total load on penetrometer, soil electrical conductivity and uv induced flourescence. Penetrometer temperature can also be recorded.

Prior to beginning a sounding, the operator manually records the zero load output of the penetrometer. At test initialization, the data logging software auto-zeros the cone tip and friction (total) loadcells. The operator then runs a shunt calibration procedure, which is recorded into the sounding log digital file. At the end of the sounding, zero readings are again manually recorded by the operator.

Zero-Error The chief quality concern in CPT load measurements is zero offset error. Zero accuracy is affected by two components - mechanical offsets and thermal offset.

Mechanical offsets are caused by dirt in seals of the penetrometer, seal compliance problems, and misalignment of components. To minimize mechanical offsets, experience and laboratory testing (Ref. 9) has shown that the stiffest (highest capacity) loadcells and in line loadcell design (subtraction type) give the most accurate results. This is because stiff loadcells minimize relative movement between components, thus minimizing ingress of dirt into the system and seal compliance problems.

The in-line design minimizes mis-alignment of components, and lessens the number of seals needed to keep soil and groundwater out of the penetrometer. STRATIGRAPHICS uses a high accuracy, high capacity in-line design for its penetrometers.

Thermal offsets are caused by frictional heating of the penetrometer as it is pushed through dense, sandy soils. Another source of thermal offset is steam cleaning of the penetrometer. A solution to thermal offset problems is to add thermal compensation elements to the penetrometer loadcell circuits, as is done by STRATIGRAPHICS. Zero readings must also be obtained prior to the penetrometer being steam cleaned.

To evaluate zero errors during penetration testing, STRATIGRAPHICS relies on two techniques:

1) zero readings before and after testing are recorded for every sounding. If significant zero drift has occurred during a sounding, the penetrometer is disassembled and cleaned, seals removed, checked, and replaced, if necessary; and

2) data recording during the sounding is done on a time basis rather than on depth basis. Thus, data are continually recorded, even while the penetrometer is not being advanced and penetrometer loadcells are unloaded during addition of sounding rods. Zero shifts can be detected by the magnitude of forces measured on the unloaded loadcells. The unload cycle data are removed from the final plot of penetration resistance versus depth through the use of software techniques.

In contrast, depth base recording, as is common in most of the penetrometer industry, does not allow for data acquisition during rod breaks. Data are only acquired while the cone is being advanced. This low cost approach to CPT does simplify data processing and field memory requirements. However, the simplification of data acquisition procedures results in loss of important field Q/A data.

Analog to Digital (A/D) Data Conversion To record and process CPT data it is necessary to convert the analog signal coming from the CPT instrument to a digital value compatible with the computerized data acquisition system. STRATIGRAPHICS utilizes an ultra high accuracy 16 bit A/D converter for this purpose.

A 16 bit analog to digital conversion provides +/- 32768 counts of digital resolution. Since the STRATIGRAPHICS penetrometers consist of two 30,000 lbs load cells, the digital resolution is better than 1 lb. By using software controllable signal conditioning gain, a digital resolution of 5 ounces (or 0.01 TSF) is achievable for work at very soft sites.

In contrast, review of equipment specifications of other CPT operators reveals use of 12 bit A/D conversion. This results in digital resolution of +/- 2048 counts. Thus, for a 30,000 lb load cell, the digital resolution is only 15 lbs, or more than an order of magnitude worse than the STRATIGRAPHICS system.

This lack of digital resolution is significant in weak soils, especially when using subtraction type instruments. Data is indicative of poor digital resolution has a characteristic step function appearance, rather than the smoothly varying actual soil response.

Depth reference Sounding depths are encoded using a bi-directional distance encoder coupled directly to the rod string. This avoids push system compliance errors. Depth encoders coupled to the thrust cylinders instead of the sounding rod string can provide erroneous information due to thrust frame and sounding rod compliance, especially during hard pushing. Depth encoders coupled to the rod string can give erroneous information if rods are allowed to become oily.

To monitor depth encoding accuracy, STRATIGRAPHICS keeps a manual record of rod string stickup prior to starting a test and at test completion. The number of rods used and rod string stickup allows computation of the exact depth of soil penetrated. These checks are used in depth Q/A procedures.